# REFINER
## AND NATURAL GASOLINE MANUFACTURER

SEPTEMBER, 1934
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**GEORGE REID**
***Editor***

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REFINER
AND NATURAL GASOLINE MANUFACTURER
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VOLUME 13, No. 9 SEPTEMBER, 1934

### Better Times

Despite the howls of some discontented refiners who have come to the state of loud talking recalcitrants through the butting of heads against conditions over which not even the "Great White Father" has control, this industry is in good condition and no sane man, viewing the situation as a whole, can be pessimistic as to its future.

There never has been a time in oil's history but that low priced crude brought on a wave of construction of small skimming plants, which served their time, faded out of the picture with higher crude prices, but returned again and usually in greater number with increased production from new flush fields and lowered prices. They have come and gone by the scores. They have come again and just as in the past many of them will shut off their fires, leave their tanks and brickwork standing as mute evidence of bad times in the refining industry. There is no malice against those who strive to make their living through the building and operation of teapot refineries—save where those operators muddy the waters of an already confusing condition through unwillingness to abide by the laws of the land. As good business people they have their rights. As hot oil merchants their rights are doubtful and their existence deplored.

This industry, like all others, sees labor, fuel costs, sources of raw material, and rapid technical advancement bring about repeated shiftings of its activities. In so far as the very small refinery is concerned its appearance inevitably follows exploitation and development of new flush fields, especially where light oil is found. Oklahoma, California, Inland Texas and the Texas Gulf Coast have seen the influx of small refiners come and go. These concerns enter such areas, or certainly should do so, with the full knowledge that their position is not long tenable and that with the passing of a very few years they must pull up stakes and follow in the wake of the drill. Some, of course, develop into established companies, grow by their versatility and initiative, and expand into concerns of national economic importance. And such types of concerns, and the men who own and run them are welcomed by the industry, respected for their ability, and receive every cooperation as they become part and parcel of the oil fraternity. The other type, and there are regretably too many of the other type, has little reason to expect either welcome or respect. It is, however, unfortunate that a small and relatively unimportant minority can be the source of sufficient confusion to darken the outlook, even temporarily, of an industry without which the world of business and pleasure can not function.

Fortunately the period of existence for the hot oil merchant is surely passing. His position is insecure and his life must be short. Even though the remainder of his existence be accompanied by howls of discontent and even to the extent of causing additional confusion in the economics of petroleum, the well established refining concern, operating honestly and aggressively in its efforts to derive a profit, should not long need to worry over the antics of its unreliable neighbor. The long time view of the industry points definitely toward continued improvement and better times.

### Italian Refineries

Recent developments in Italy indicate that the long talked of activity in refinery and cracking plant construction will actually get under way early in 1935. National and international petroleum interests operating or proposing operations in Italy, after months of waiting saw the new petroleum decree approved by the Council of Ministries late in June. Following publication of the decree, upon which it becomes law, it must be then followed by interpretation and application, including actual granting of import and refining concessions which are slated to become effective January 1, 1935.

In so far as plant capacity and number of plants to be permitted, as well as their location and type, the Italian government stresses first the matter of national defense, second national revenue, and apparently concerns itself with an economical or

profitable oil industry last. The government, however, has intimated that it has no intention of allowing refinery construction and expansion to be developed beyond what it considers suitable for requirements. Regulations requiring domestic refining will certainly bring about marked construction activity in that country.

When and if the licenses are granted and contingent upon requirements and restrictions, a number of concerns are ready to begin construction work, both in improvements and new refineries. Socony-Vacuum Corporation, which purchased control of the BENIT cracking plant at Naples, is reported planning to rebuild and convert the plant into a complete refinery. The AGIP which is controlled by the government, has purchased the DISCA cracking plant at Venice, and is reported planning reconstruction into a complete plant; also reported planning modernization of its plant at Fiume to a complete plant, and may also erect a new refinery. The SIAP (Standard Oil Company (New Jersey) subsidiary) is reported planning enlargement of its refinery at Trieste and considering establishment of another plant on the west coast. Nafta (Royal Dutch-Shell group) is said to be planning reconstruction of its Spezia cracking plant and erection of a new one at a location which must be approved by the Supreme Council of Defense. Petrolea (Soyusnefteexport) is also reported planning a refinery to handle its business in refined products except bunker fuel. Among others which have filed applications for concessions are Gulf Oil Corporation (Union Importatori Lubrificanti) and The Texas Company. All locations are to be approved by the Supreme Council of Defense following the recommendations of the Ministry of Corporations and the Ministry of Finance.

All indications now point to an active construction program in Italy as soon as the oil companies can secure complete information as to what their concessions will permit.

***Synthetic Products*** Prospects of a petroleum chemical industry are rapidly forming into a phase of petroleum refining that is almost daily assuming a position of commercial importance and the progress of a chemical industry based on petroleum during recent years has been most gratifying. New products of petroleum derivation have been making their appearance in steadily increasing number and are fast assuming a more intimate relation to our daily lives. Not many of those engaged in the petroleum refining industry are able to realize the present relative importance of developments of this type although virtually all of the important new products have been mentioned from time to time in the technical and trade press. It is only when such information is correlated and discussed in detail, with many of the numerous products mentioned both as to their method of manufacturer and uses, that the importance and rapidity of the developments are fully realized. Dr. Carleton Ellis, Industrial Research Consultant, in his paper before the recent New York meeting of the American Institute of Chemical Engineers brings much of this information down to date in a most interesting fashion. Because of the growing importance of this phase of the industry's activities, Dr. Ellis' paper is presented in this issue. Page 337.

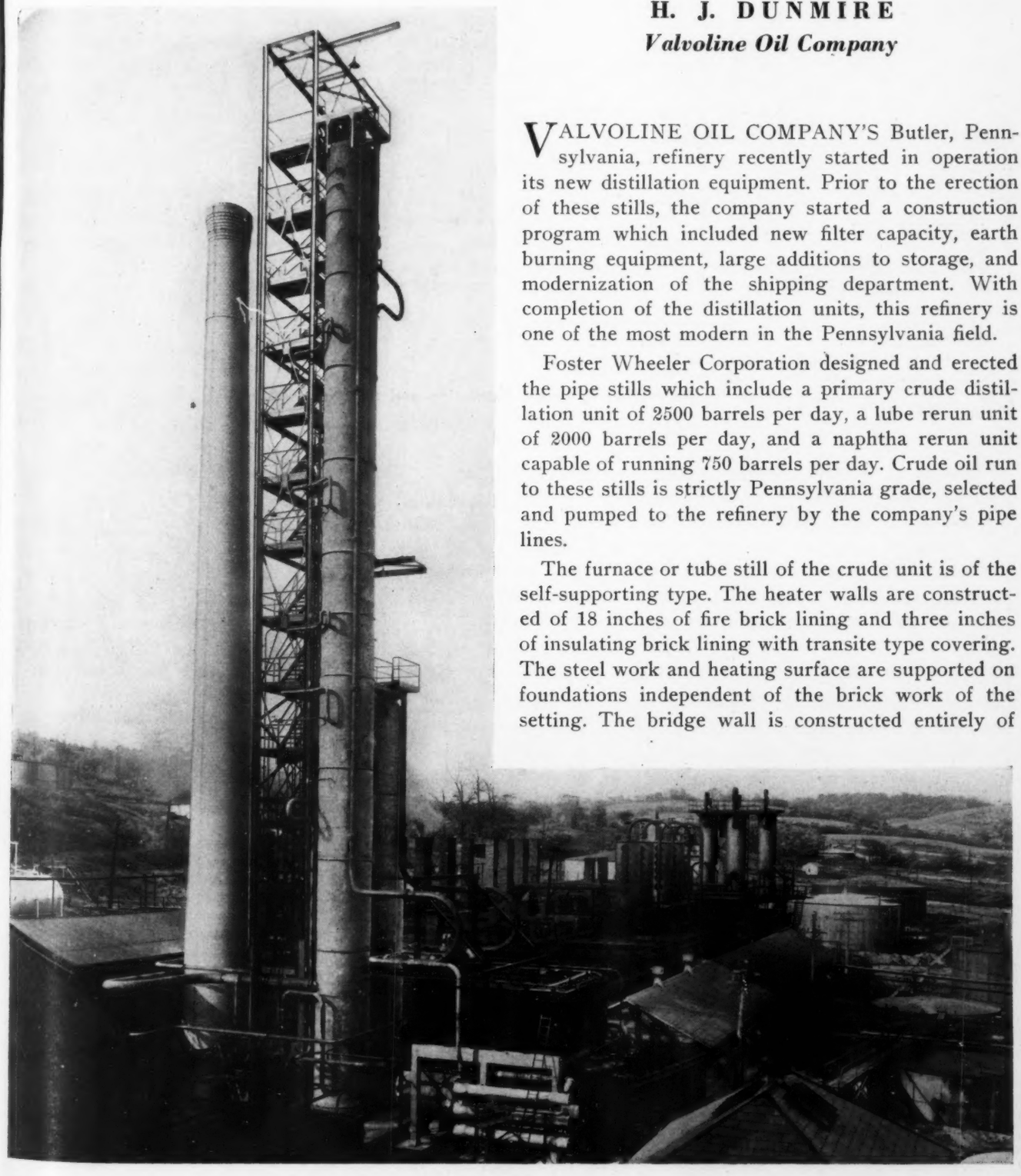
***Propane Dewaxing*** From a general chemical and physical viewpoint the following important factors are involved in the manufacture of lubricating oils; selection of fraction of proper boiling range, removal of asphaltic and/or coloring materials; removal of naphthenic materials (concentration of paraffinics) and removal of waxes. In a recent paper before the California Natural Gasoline Association, "Use of Propane in Lubricating Oil Refining," U. B. Bray, C. E. Swift, and D. E. Carr, of Union Oil Company of California, presented much interesting and valuable data on the modern process of propane dewaxing and deasphalting as employed by that company. The paper, together with several views of the recently completed plant at Oleum, California, is presented in this issue through the courtesy of the association and Union Oil Company. Page 333.

***Stabilizer Systems*** In expansion and extension of this subject "Elements of Stabilization," I. N. Beall, Chemical Engineer, this month contributes Part 3 of his series of articles. This series is being prepared with the idea of making the technical and engineering phases of the subject more easily adapted to practical application. In the present discussion methods are described whereby maximum yields of substantially pure hydrocarbon fractions may be produced from natural gas, or other gases, including separation of methane, ethane, propane, isobutane and butane and the pentanes. Part 4 of the series will appear in an early issue. Page 346.

# *Valvoline Oil Company's*

# New Distillation Units

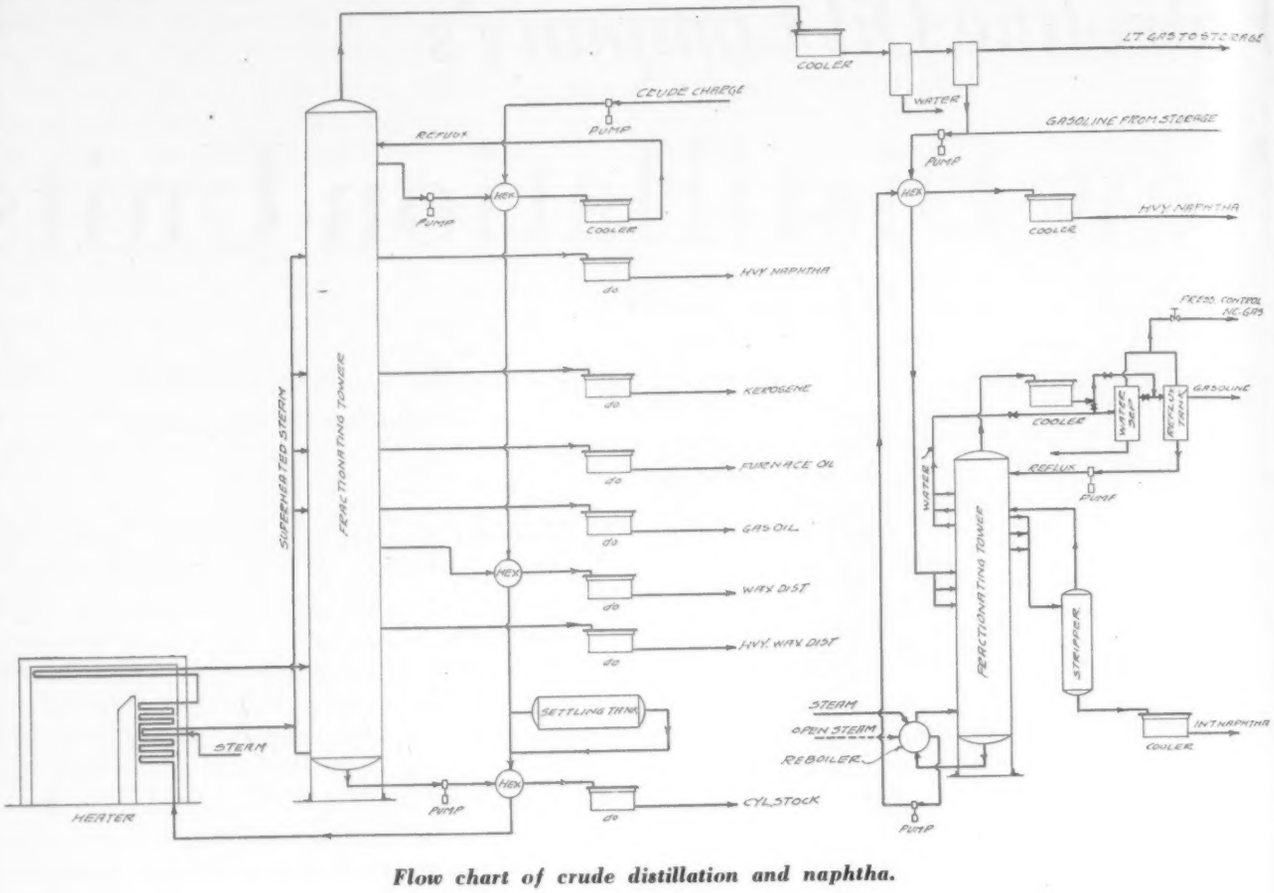
**H. J. DUNMIRE**
***Valvoline Oil Company***



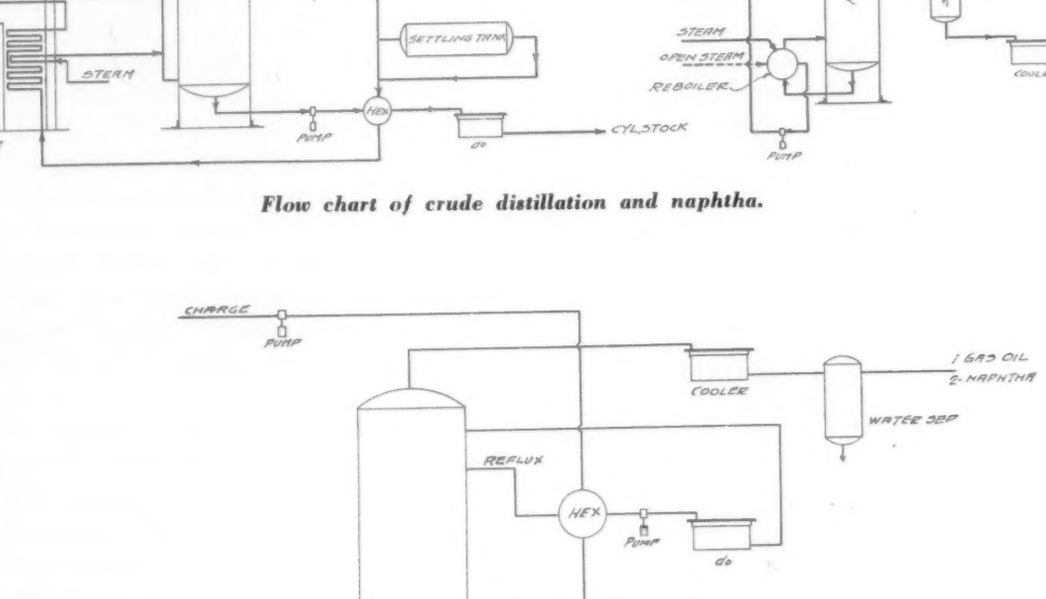
VALVOLINE OIL COMPANY'S Butler, Pennsylvania, refinery recently started in operation its new distillation equipment. Prior to the erection of these stills, the company started a construction program which included new filter capacity, earth burning equipment, large additions to storage, and modernization of the shipping department. With completion of the distillation units, this refinery is one of the most modern in the Pennsylvania field.

Foster Wheeler Corporation designed and erected the pipe stills which include a primary crude distillation unit of 2500 barrels per day, a lube rerun unit of 2000 barrels per day, and a naphtha rerun unit capable of running 750 barrels per day. Crude oil run to these stills is strictly Pennsylvania grade, selected and pumped to the refinery by the company's pipe lines.

The furnace or tube still of the crude unit is of the self-supporting type. The heater walls are constructed of 18 inches of fire brick lining and three inches of insulating brick lining with transite type covering. The steel work and heating surface are supported on foundations independent of the brick work of the setting. The bridge wall is constructed entirely of




***Flow chart of crude distillation and naphtha.***



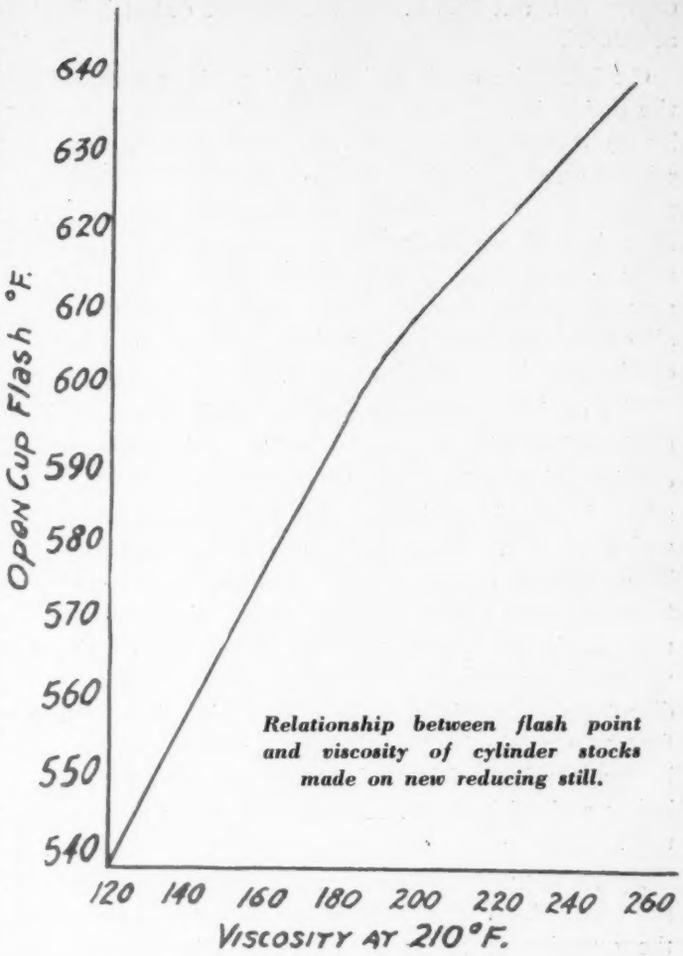

***Flow chart of lube rerun unit.***

fire brick and is air cooled. Two important features are incorporated in the design to prevent decomposition of the crude oil. First, the inside diameter of the tubes is selected to permit evaporation in the tubes of the heater, thus supplying the latent heat as the boiling point is reached and thereby reducing the differential between the temperature of the heater outlet and that at the bottom of the tower to 10° or 15° F. In this way the mixture of liquid and vapor attains a very high velocity in the heater tubes and the latent heat required to complete evaporation in the bottom of the tower is reduced to a minimum. This feature is important when handling high grade Pennsylvania crudes which are particularly sensitive to temperature. The second feature incorporated allows the length of travel through the tubes of the heater to be materially reduced by extending the heating surface on the outside of the tubes in the convection bank. Gill rings are used; the rings being larger toward the bottom of the convection bank. The radiant section tubes are located under the roof of the heater and are arranged in two rows of staggered tubes, one row above the other; the final passage of the oil being through the upper row.

The fractionating tower is 133 feet high by six feet six inch diameter and contains 64 trays. Each side stream from the tower is controlled by a wier type of proportioning valve which, once set, will withdraw side streams to specifications irrespective of the quantity charged to the still. Internal stripping sections are used in the tower. The use of this type of stripper permits removal of the light ends without loss of heat by radiation from outside vessels and pipe connections. The exhaust steam from the pumps is superheated in a superheater section located in the convection bank of the furnace. This superheated steam is available for stripping in all sections of the tower.

Crude oil from storage is pumped through the liquid reflux and wax distillate exchangers. It then passes through a five by 25-foot settling drum where water, sand, or other impurities carried by the crude are settled out under pressure and at about 250° F. temperature. The charge then goes through the bottoms exchanger and enters the furnace at about 350° F. When running for 150 viscosity steam refined stock, a transfer temperature of 650°-660° F. is used.

The crude unit was designed to manufacture steam refined stocks ranging in viscosity from 80° at 210° F. to 280° at 210° F. The specially designed steam stripping section in the bottom of the tower permits of exceptionally high flash tests on the steam refined stock when running to a given viscosity. Chart 1 shows a plot of flash versus viscosity. These figures are average obtained over several months operation. It will be of interest to note that stream samples of 250° viscosity at 210° F. steam refined stock showed flash tests of 645° F.




***Relationship between flash point and viscosity of cylinder stocks made on new reducing still.***

Besides the steam refined stock six side streams and an overhead cut may be drawn simultaneously from the tower. These streams from top to bottom are light gasoline overhead, solvent, kerosene, 300 oil distillate, gas oil, pressable wax distillate, and heavy wax distillate. The heavy wax distillate cut is taken off only when the viscosity of the bottoms runs higher than 170° at 210° F.

The lube rerun unit contains many of the features found on the crude unit. The furnace is of similar construction with the steam superheater section located in the convection bank. Streams withdrawn include an overhead cut, two side streams, and a lube bottoms stream. Superheated steam is used in the various stripping sections and Wier type valves control the two side stream withdrawals. The tower is five feet in diameter by 57 feet high. Liquid reflux is withdrawn from the tower and used to preheat the charge before being pumped back to the top of the tower.

The charge to this unit is preheated by pumping through the reflux and bottoms exchangers. When charging pressed wax distillate, the charge enters the furnace at about 300°F. When running to 150 viscosity neutral bottoms, a transfer temperature of 665°F. is used. The transfer temperature used when running to 200 neutral bottoms is about 680°F. When charging bright stock solution, the preheated

charge is about 290°F. and the furnace outlet is held at 600°F.

On both the crude unit and the lube rerun unit, the end point of the overhead product is controlled by maintaining the top tower temperature constant with the use of liquid reflux pumped from the upper trays and cooled before being returned to the tower. By using this method of reflux a vapor exchanger is not necessary and all exchangers are placed near the ground level where they are easily accessible. It is also possible to use a fraction from the overhead condensate for reflux if desired.

The naphtha rerun unit was erected to operate in conjunction with the crude unit. This equipment consists of a main tower 57 feet high by three feet seven inches in diameter, a side stripping section, and a reboiler. Three cuts are made simultaneously. The naphtha charge can be pumped from storage tanks or taken direct from the crude unit water separator tank, or, by means of a bypass, the overhead vapors from the crude unit tower may be passed direct to the naphtha unit. When charging by the first two methods, the charging stock is preheated by exchanging with naphtha bottoms from the reboiler and with steam refined stock from the crude unit.

By varying the specifications of the naphtha charging stock, by changing the top tower temperature, the quantity of steam used in the side stripper, or the temperature of the reboiler, a wide variety of naphthas may be manufactured. Included in this group are such commercial grades as Stoddard solvent, rubber solvent, V. M. & P. naphtha, turpentine substitute, and others.

All three units are equipped very completely with automatic control instruments. Air-actuated rate-of-flow meters control the rate of oil charged to each unit. Temperature control instruments control the amount of reflux necessary to hold the top tower temperature constant on each unit. A four-point recording pyrometer records top of tower and flash zone temperatures of both the crude and lube rerun units. A two-point recording pyrometer gives a record of the top tower and reboiler temperatures on the naphtha rerun unit. The tube still inlet and outlet temperatures of both furnaces are recorded on two two-point pyrometers. Air-actuated liquid level controllers assure a constant level in the bottom of each tower. The furnace temperatures of both crude and lube rerun units are automatically controlled. Steam flow meters measure the superheated steam delivered to the bottoms stripper in both the crude and lube rerun towers. Each steam line to all other stripping sections is equipped with steam flow meters to record the steam flow. Temperatures throughout the furnaces and towers of the three units may be obtained instantly by the use of a 24-point indicating pyrometer with rotary switches. Make-up steam is supplied to the exhaust line through an air-actuated exhaust steam pressure controller. All pumping is steam driven.

The accompanying table shows specifications of some of the products which have been manufactured on the crude oil and lube rerun units. These tests, while showing somewhat the flexibility of the steam refined stocks, do not give a true picture of the variety of oils which may be obtained from the other streams. By changing the reflux ratio, the temperatures, and/or the steam input into the various sections of the towers, the specifications of the side and overhead streams may be materially changed.

## Average Laboratory Tests of Oils Manufactured

| | Grav. | Flash | Fire | Vis. 100 | Vis. 210 | Cold Test | Con. Carb. | Pensky Martin | IBP | EP. | Color |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Crude Unit:** | | | | | | | | | | | |
| Gasoline | 61.5 | .... | .... | .... | .... | .... | .... | .... | 125 | 375 | 25+ |
| Naphtha | 51.5 | .... | .... | .... | .... | .... | .... | .... | 250 | 395 | 25+ |
| 45 Kerosene | 45.0 | 175 | 200 | .... | .... | —20 | .... | .... | 365 | 585 | 25+ |
| 47 Kerosene | 47.0 | 165 | 190 | .... | .... | —20 | .... | .... | 360 | 530 | 25+ |
| Furnace Oil | 40.0 | 230 | 300 | 44½ | .... | 35 | .... | .... | 380 | 645 | .... |
| Fuel Oil | 38.1 | 280 | 330 | 52 | .... | 50 | .... | .... | 384 | 725 | .... |
| Wax Distillate | 35.5 | 325 | 405 | 75 | 44½ | 70 | .... | .... | .... | .... | 1½ |
| 125 Vis. SRC | 27.5 | 545 | 615 | .... | 125 | 55 | 1.46% | 515 | .... | .... | .... |
| 150 Vis. SRC | 26.8 | 570 | 640 | .... | 150 | 35 | 2.0% | 550 | .... | .... | .... |
| 180 Vis. SRC | 26.5 | 600 | 670 | .... | 181 | 35 | 2.32% | 575 | .... | .... | .... |
| 195 Vis. SRC | 26.4 | 610 | 680 | .... | 195 | 35 | 2.6% | 580 | .... | .... | .... |
| 210 Vis. SRC | 26.2 | 615 | 685 | .... | 212 | 35 | 2.8% | 585 | .... | .... | .... |
| 225 Vis. SRC | 26.0 | 620 | 695 | .... | 228 | 35 | 3.0% | 595 | .... | .... | .... |
| 250 Vis. SRC | 25.8 | 640 | 705 | .... | 245 | 35 | 3.0% | 615 | .... | .... | .... |
| **Rerun Unit:** | | | | | | | | | | | |
| Fuel Oil | 37.4 | .... | .... | .... | .... | .... | .... | .... | 326 | 706 | Lt. Straw |
| 1st. Side 60 Vis | 35.5 | 335 | 380 | 60 | .... | 25 | .... | .... | .... | .... | 2 |
| 2nd. Side 100 Vis | 32.8 | 395 | 450 | 100 | .... | 25 | .... | .... | .... | .... | .... |
| Bot. 150 Neut | 31.7 | 425 | 490 | 150 | .... | 20 | 0.5% | 400 | .... | .... | 4 |
| Bot 180 Neut | .... | 435 | 500 | 180 | .... | 20 | 0.60% | 410 | .... | .... | 4 |
| 85 Vis. B. S. Wash | .... | 480 | 545 | .... | 85 | 20 | .80% | 460 | .... | .... | 8 |
| 125 Vis. Br. Stock | .... | 545 | 615 | .... | 125 | 20 | 1.0% | 520 | .... | .... | 8 |
| 145 Vis. Br. Stock | .... | 570 | 640 | .... | 145 | 20 | 1.2% | 545 | .... | .... | 8+ |

Pictures Courtesy Union Oil Company of California.

# USE OF . . .

# Propane in Lube Oil Refining

**ULRIC B. BRAY, CLAUDE E. SWIFT and DONALD E. CARR**

***Union Oil Company of California, Los Angeles***

***Before the California Natural Gasoline Association***

THE problem of producing superior lubricants from asphaltic or mixed-base crudes has been given wide attention during recent years, and as a result several new processes and improvements in existing methods of manufacture have been devised. The purpose of this paper is to present some outstanding uses of propane in lubricating oil manufacture which have been studied in the laboratories of Union Oil Company of California over a period of the last five years*. It will be shown that propane may be used advantageously:

1. To de-asphaltize asphaltic or mixed-base residua,
2. To dewax residual or distilled oils,
3. As a diluent in acid and clay treating,
4. In combination with selective refining solvents.

*Patents have been applied for on various features of these processes.

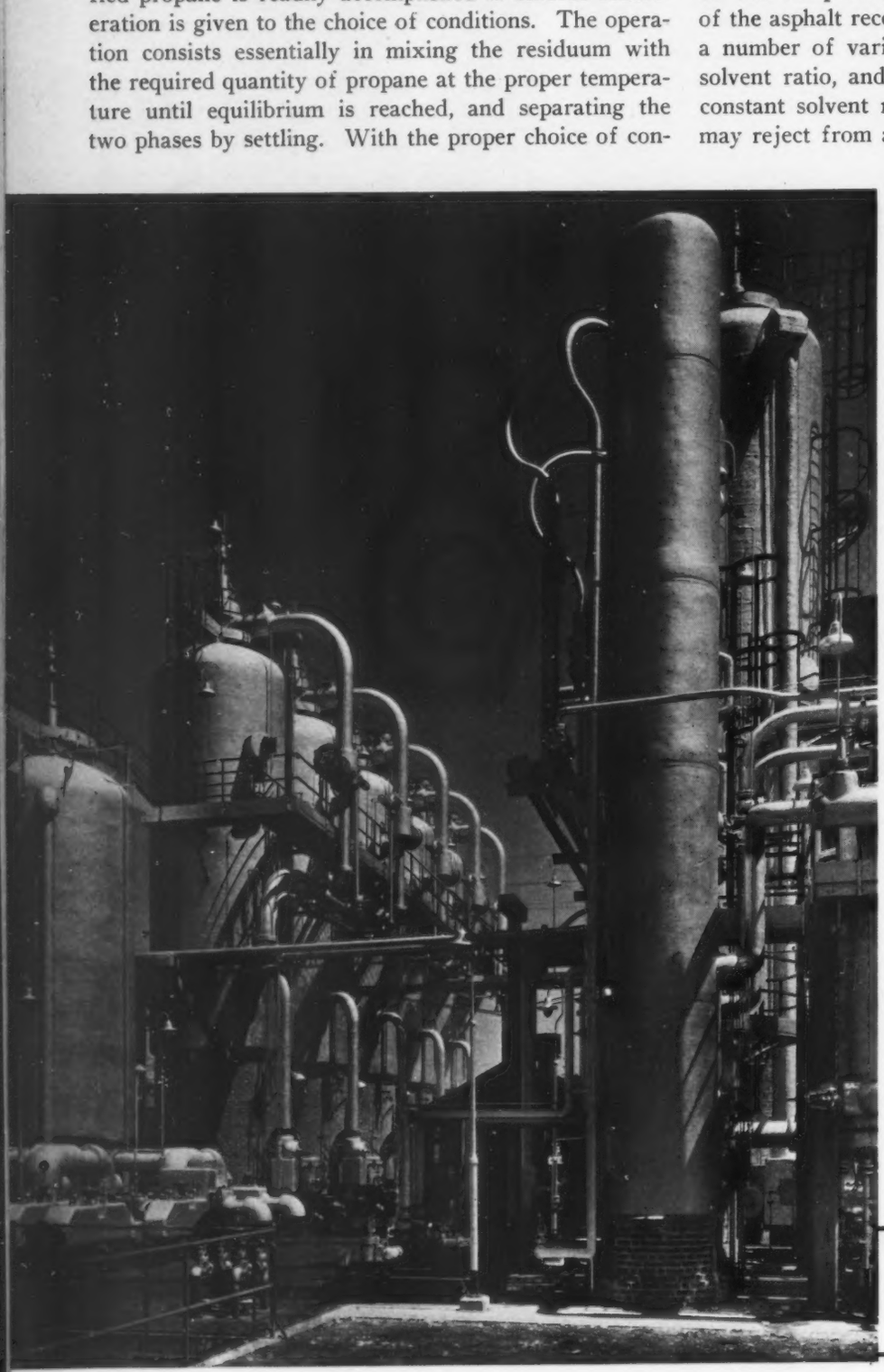
The use of propane to separate lubricating oils from asphaltic residues in contrast to distillation makes possible the recovery and utilization of the lubricating fractions of extremely high molecular weight which are most difficult to distill without decomposition. The ease of recovery of these residual lubricants in their natural state eliminates to a considerable extent the necessity of selecting crudes of low asphalt content for the production of residual bright stocks and cylinder stocks. The recovery of these residual oils also furnishes stocks for the production of oils of low viscosity-

temperature susceptibility, because some of these high-molecular-weight residual fractions exhibit the flat-test viscosity-temperature curves of any oils obtainable from a given crude.

The removal of asphaltic material by means of liquefied propane is readily accomplished if careful consideration is given to the choice of conditions. The operation consists essentially in mixing the residuum with the required quantity of propane at the proper temperature until equilibrium is reached, and separating the two phases by settling. With the proper choice of conditions, the propane phase—consisting of a solution of the lubricating oils—is obtained as a clear, transparent, super-natant liquid; while the asphalt phase can be brought down as a heavy, viscous liquid containing sufficient dissolved propane to permit ready transfer by pumping. The process is capable of continuous operation with simple equipment.

The extraction of residual oils with low-molecular-weight hydrocarbons, i.e., ethane, propane, the butanes, and mixtures of these, leads to a better understanding of the composition of asphalt, because the character of the asphalt recovered from the process depends upon a number of variables—including solvent composition, solvent ratio, and temperature. For example, using a constant solvent ratio of say 5 to 1 at 80° F., butane may reject from a certain residuum a small amount of high-melting asphalt; propane will reject a larger quantity of lower-melting asphalt having the general characteristics of the usual steam-blown product; whereas, pure ethane will reject a very large proportion of the residuum as a heavy asphaltic liquid. Table 1 and Figure 1 illustrate the effect of employing the different solvents on a residuum obtained from a typical low-cold-test crude topped to remove lighter fractions through the gas-oil range. For comparison, tests using pentane and hexane are shown. It may be stated that substantially pure propane comes the nearest of all the solvents to segregating the residuum into lubricating oil and asphalt of usual characteristics.

Considering the efficiency of asphalt separation with decreasing average molecular weight or decreasing average boiling point of the solvent, we find a quite regular progression of improvement in going from hexane to butane; but, upon going to lower average boiling points and molecular weights, we find a surprising increase in efficiency of asphalt precipitation—far greater than would be expected on the basis of the results obtained with butane and the higher-molecular-weight solvents. In going from butane to propane, for example, the increase in asphalt precipitated is about two and a half times as great as that to be predicted on the basis of the results obtained with hexane, pentane, and butane on the same residuum (Figure 1). The abrupt increase in efficiency of as

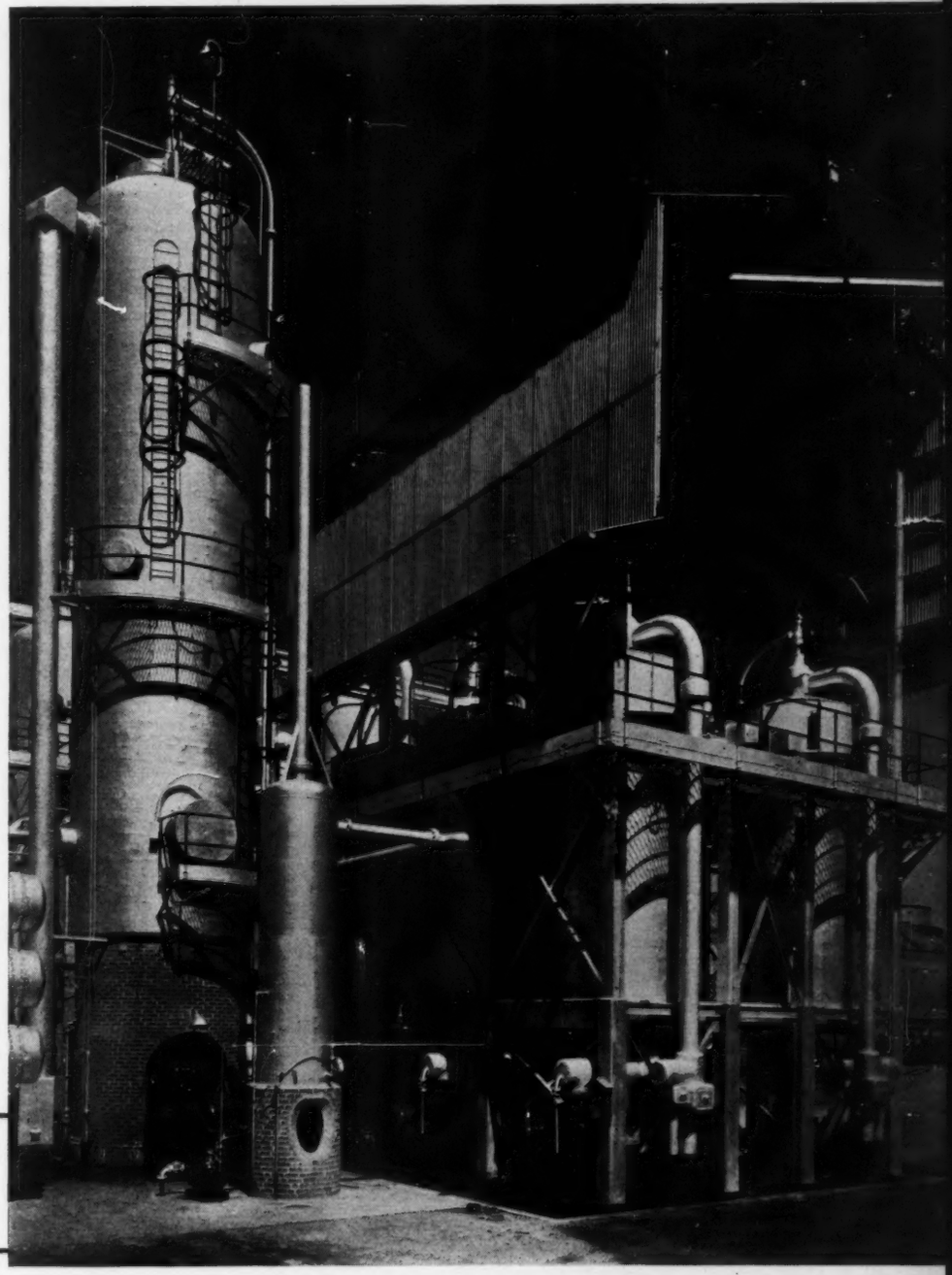


***Propane recovery system on the right and battery of chillers at left.***

phalt precipitation in going from butane to propane is also reflected in the character of the extracted oils recovered. The oil recovered by butane from a topped asphaltic residuum is extremely viscous, low in A.P.I. gravity, and dark brown in color, due to the large proportion of asphalt not precipitable by butane. In distinct contrast to this is the bright green appearance of propane-extracted oils, which have lower viscosities, higher A.P.I. gravities, and lower carbon residues, due to the practically complete precipitation of asphaltic bodies.

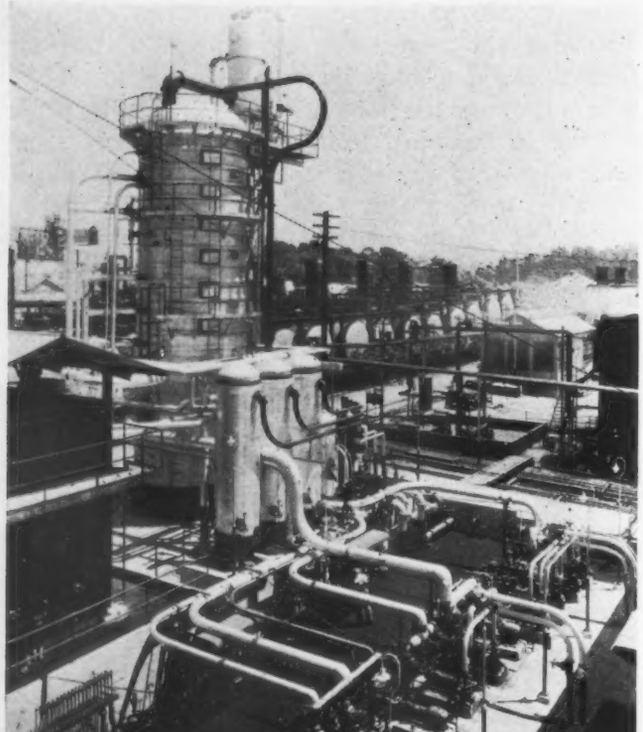
Extraction with pure ethane follows the break in the asphalt-precipitation curve by propane, and, therefore, rejects a large portion of hydrocarbons which in reality come under the classification of lubricating oils. In other words, pure ethane proceeds farther than is usually desirable in separating the heavy fraction from the lighter lubricating fractions; but very unusual residual oils of low viscosity, which are extremely low in color and carbon residue and of high flash point, can be produced for special purposes by the use of ethane on a well-fractionated residuum.

Confining attention to a solvent of definite composition at a given temperature, the results obtained in the separation can be varied over a certain range by the choice of solvent ratios. With propane at 80° F., for example, and a topped residuum containing 15 to 20 percent of asphalt, it is found that the addition of small amounts of propane to the residuum up to a certain proportion serves only to reduce the viscosity in a manner analogous to the addition of any light solvent. Further addition of propane in a small amount beyond a critical ratio causes separation into two phases, with the asphalt phase occurring as a heavy, viscous liquid in comparison to the extremely fluid propane phase. The asphalt phase recovered at this point shows, after removing solvent, a low melting point due to incomplete removal of heavy oils, and the oil phase recovered may contain several percent of asphalt. If the propane ratio is increased, the asphalt phase becomes more viscous in character; and, upon separating and evaporating the solvent, the melting point is found to have increased—due to more complete transfer of the heavy oils into the propane phase; and the oil recovered from the propane phase simultaneously shows a smaller asphalt content. Eventually a point is reached where further additions of propane produce no further perceptible improvement in fractionation of oil and asphalt, which then appears as finely-divided slurry settling from the propane solution. For convenience this ratio of propane, beyond which there is practically no improvement, is called the optimum propane ratio.



*Battery of wax filters of type used in connection with the propane solvent process.*

Figure 2 illustrates the variation in voscosity of the extracted oil with proportions of ethane or butane to propane. The changes in other properties of the ex-



Courtesy—Foster Wheeler Corporation.

***Vacuum distillation unit at Union Oil Company's Oleum, California, refinery used to secure closely fractionated lubricating oil cuts of desired grades.***

tracted oils, such as gravity and carbon residue, are parallel. The butane used varied from substantially equal proportions of *iso*butane and normal butane to substantially all *iso*butane as the butane content decreased.

Figure 3 illustrates the effect of changing the volume ratio of propane on the viscosity and gravity of the oil recovered.

It should be pointed out that the residuum chosen for these illustrations is of the purely naphthenic type, containing practically no paraffin wax; but, in the case of wax-bearing residua, the wax is completely soluble in the propane at 80° F., and behaves as a corresponding amount of lubricating oil at that temperature.

As an aid in establishing a more complete picture of propane-de-asphaltizing, we may focus attention upon the material insoluble in pure propane or, practically speaking, in propane containing only a very small concentration of oil, and designate this material as "propane-insoluble asphalt." All asphalts recovered by propane from the same residuum, when using a smaller propane ratio, will consist of propane-insoluble asphalt, plus an amount of oil depending on the solvent ratio. With this understanding, it is readily possible to construct a triaxial phase-rule diagram showing in succinct form the composition of the phases to be expected from the asphaltic or mixed-base residuum when it is contacted with liquid propane in any proportion.

Experimentally, this is done by carrying out a series of careful extractions with varying propane ratios—on a given residuum; measuring the propane content of both oil and asphalt phases; and re-extracting the oil phase with a high propane ratio, in order to determine the asphalt passing into the oil phase with the initial solvent ratio. In re-extracting the oil, it is preferable to reduce the oil first by vacuum or steam distillation, in order to concentrate the asphalt in the bottoms—thereby bringing within reason the amount of propane necessary to obtain extreme selectivity. Having prepared the equilibrium chart from the phase analyses on a given reduced asphalt-bearing crude, we may predict what will happen when propane is mixed with the same crude topped to different degrees and, to a large extent, even the behavior of dissimilar crudes. With a different sample of residuum, it is necessary only to determine the asphalt-oil content of the material, i.e., locate the point on the asphalt-oil side of the triangle corresponding to the residuum itself, by performing an analytical extraction with an extremely high propane ratio, such as 20 to 1. The point on the asphalt-oil side of the triangle corresponding to the composition of the residuum in question is next joined with the propane vertex by means of a straight line; and we may then predict, for example, the critical proportion of propane necessary to afford a separation into two phases by taking the composition at the intersection of the straight line with the solubility curve. The prediction of optimum propane ratio for selectivity is also readily made.

In Figure 4, a phase-rule diagram, constructed from the results of propane analyses on Poso Creek residuum topped with steam to 66 sec. Saybolt Universal viscosity at 100° F. on the overhead stream, is presented. While some variation from the solubility curve is to be expected with crudes of widely different characteristics, the diagram has proved to be of considerable value in obtaining a practical estimate of the results to be obtained with a large number of residua of different characteristics. For example, from the extraction of McKittrick residuum topped to 81 sec. Saybolt Universal viscosity on the overhead stream, with 15 volumes of propane, the asphalt content of the latter was approximately determined, and the propane ratio necessary for separation into two phases was calculated by measuring the relative distances from the propane vertex and the McKittrick composition point to the intersection of a straight line between these points with the solubility curve. This calculated ratio was 0.7 to 1, while the ratio found by actual determination was 0.65 to 1.

The effect of lowering the temperature with a given propane ratio is to decrease the solubility of the asphaltic material in the propane-oil phase. Figure 5 illustrates the effect of variation in the temperature of propane extraction, on the gravity and viscosity of the

(*Continued on page* 353)

# *Prospects of a*
# Petroleum Chemical Industry

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**DURING recent years there has been a steady trend toward the establishment of a chemical industry dependent upon petroleum as a source of raw material. With the large number of possibilities offered by a multitude of already available substances, future developments may be even more surprising. In the accompanying paper presented by Carleton Ellis before the recent meeting of the American Institute of Chemical Engineers, New York, the author outlines this trend and discusses the chemistry of production of a large number of products derived from petroleum. Among such products are the alcohols, acetone and other ketones, ethylene dichloride, Thickol, DuPrene, Vinylite, Carbitol, butter substitute flavoring, resins, drying oil substitutes, solvents, wax acids for making synthetic fats and making soaps, especially hard water types, denaturants such as Aldehol and Alcotate; Paraflow, Pentasol and Pentacetate, insecticides, deodorants such as Cal-odorant, sulfonic acids and others. The prospects of an even more extensive chemical industry based on petroleum is discussed. The author's references to the literature have been omitted, in this instance and the reader is referred to the original paper or to Dr. Ellis' latest book "The Chemistry of Petroleum Derivatives" for more thorough study.**

THE purpose of this paper is not to enumerate all the chemical possibilities through the conversion of petroleum material, since the number of these proposals is legion. However, it is clear that the real developments involving various chemical engineering problems have fallen to the gases from petroleum, rather than to the liquid and high-boiling constituents. One reason for this is that the gases afford a means of extracting concentrates or even pure hydrocarbons such as methane, ethane, propane, butane, ethylene, propylene, butylene, etc. For making useful products, such concentrates are better adapted than difficultly separable mixtures of higher hydrocarbons of undetermined constitution.

Crude petroleum consists essentially of a mixture of hydrocarbons of varying degrees of molecular complexity. In addition there are present smaller proportions of compounds of oxygen, nitrogen, and sulfur, together with mechanically held gas, water, or clay and other inorganic substances, either in solution or colloidal suspension. The hydrocarbons may range from the simplest to those having molecular weights greater than 1000, and include not only paraffins and cyclo-paraffins (naphthenes) but also aromatics and polynuclear aromatics and cycloparaffins. The occurrence of unsaturated hydrocarbons, prior to distillation, has been the subject of much controversy. If such hydrocarbons are present in crude petroleum, the proportion must be exceedingly small.

It has been calculated that a hydrocarbon represented by the comparatively low number of 20 carbon atoms has 366,319 possible isomers (or 3,395,964, if stereoisomers are included). The difficulties involved in separating such a complex mixture as petroleum are evidently very great. Although many individual hydrocarbons have been isolated by laborious fractional distillation and crystallization methods from petroleum fractions, particularly natural-gas gasoline and both straight-run and cracked gasolines, the gaseous hydrocarbons are the only compounds readily separated. Also, some of the gaseous hydrocarbons are often more reactive than their higher boiling analogs. At the present time, therefore, these gaseous fractions are the more promising crude materials for chemical conversion.

The first real chemical industry founded on petroleum was probably the production of alcohols by reaction of the olefins of cracking gas with sulfuric acid.

### CRACKING GAS

Industrial cracking processes are, for the most part, concerned with the conversion of high-boiling distillates into low-boiling liquids of the gasoline type. The utilization of the large quantity of lower hydrocarbon by-products (cracking gas) from this procedure is already exerting a tremendous influence on the development of industrial organic syntheses. It has been estimated that the volume of this gas is of the order of 250 billion cubic feet annually.

The composition of cracking gas is dependent chiefly upon the temperature of cracking. The so-called liquid-phase operations [employing temperatures below 500° C. (932° F.) and pressures up to 1000 pounds per square inch] yield a gas containing a fairly large proportion of paraffin hydrocarbons together with a relatively small content of olefins. Of the latter, propylene is apparently the predominating unsaturated hydrocarbon. Vapor-phase processes (operating at higher temperatures and at lower or

atmospheric pressures) give a cracking gas having a large proportion of ethylene in addition to propylene and other olefins. Diolefins, particularly butadiene, are also present in appreciable quantities (one per cent or more).

Prior to its utilization, cracking gas may be subjected to compression and absorption operations, thereby dividing it into at least two main fractions: (a) gaseous olefins, consisting of ethylene, propylene, and butylenes, and (b) liquid olefins, consisting of amylenes, hexylenes, and higher olefins. Further separation of these two main fractions is, of course, possible.

Gaseous and low-boiling hydrocarbons may also be obtained from cracked distillates. Cracked naphtha —e. g., containing fixed gases soluble at the temperature and pressure of the condensing equipment on the cracking coils—is fed to a tower which is called a "debutanizer." About 30 percent of the most volatile components are taken overhead in this tower, and substantially all are condensed. This volatile overhead cut is then fed to a second tower which is called a "stabilizer." Here a sharp fractionation is made between propane and lighter hydrocarbons, and butane and less volatile hydrocarbons.

The analysis of the overhead gas and residue (bottoms) from pressure cracking stills varies with variations in cracking conditions but, by way of illustration, is approximately as follows:

| Overhead from Debutanizer | % by vol. | Bottoms from Stabilizer | % by weight |
|---|---|---|---|
| Methane and fixed gas | 3 | Isobutane | 4 |
| Ethylene | 4 | Isobutene | 4 |
| Ethane | 21 | 1-Butene | 3 |
| Propylene | 16 | *n*-Butane | 13 |
| Propane | 49 | 2-Butene | 6 |
| Isobutane | 2 | *n*-Pentane | 20 |
| | | Amylenes | 13 |
| | | *n*-Hexane | 15 |
| | | Hexenes | 9 |
| | | Heptane and higher | 13 |

With modern pressure-fractionating equipment, the separation of liquefied petroleum gases into propane, *n*-butane, and isobutane is a relatively simple procedure which is being conducted in many plants. The resulting butane is employed as a carbureting agent, and propane, in addition, is used in refrigerating equipment. Liquefied isobutane or propane are extracting agents for the refining of resins and gums or oils and asphalts.

## ALCOHOLS

The conversion of olefins to alcohols is accomplished by absorption in sulfuric acid. The alkyl sulfuric acids which are formed as intermediates are then hydrolyzed to alcohols. Except with ethylene, secondary and tertiary alcohols are thus obtained. The reactions for ethylene and propylene may be represented by:

$$H_2C{=}CH_2 \xrightarrow{+H_2SO_4} CH_3CH_2SO_4H \xrightarrow{+H_2O} CH_3CH_2OH + H_2SO_4$$

$$CH_3CH{=}CH_2 \xrightarrow{+H_2SO_4} \begin{matrix} H_3C \\ H_3C \end{matrix}\!\!>\!CHSO_4H \xrightarrow{+H_2O} \begin{matrix} H_3C \\ H_3C \end{matrix}\!\!>\!CHOH + H_2SO_4$$

The products from ethylene and propylene are exclusively ethyl and isopropyl alcohols, respectively. With butylenes and higher olefins, either secondary or tertiary alcohols, or a mixture, are obtained, as well as a certain proportion of polymers.

Possibly the most important factors connected with this method of manufacturing alcohols are the temperature and concentration of the acid, both of which determine to a large degree the proportion of olefin converted to alcohol or to polymers. With ethylene, temperatures as high as 60° to 80° C. (140° to 176° F.) may be employed for other olefins (propylene and higher) the temperature is generally kept at 20° to 30° C. (68° to 86° F.) or lower. Ethylene is absorbed in 96 to 100 percent acid, propylene in 90 to 94 percent acid, higher straight-chain olefins (e. g., 1- and 2-butene and 1- and 2-pentene) in 80 to 90 percent acid, and isoolefins (isobutene and isopentene) in 60 to 70 percent acid. Reaction between the olefin and acid, especially with gaseous olefins, is increased and facilitated by increased pressures and agitation in the presence of a liquid (e. g., a high-boiling petroleum fraction) which is a solvent for the olefin but which is immiscible with the acid. Many different catalysts have been proposed which not only aid absorption of the olefin by the acid but also reduce losses from polymerization.

Alcohols which are produced on a commercial scale by the sulfuric acid process are ethyl, isopropyl, *sec*- and *tert*-butyl, *sec*-amyl, and *sec*-hexyl.

The use of the two lower alcohols, ethyl and isopropyl, as solvents and in pharmaceutical preparations is too well known to require further comment. Unique uses for higher secondary and tertiary alcohols are in the purification of paraffin slack wax, in paint-removing compositions, and as fuels for internal combustion engines. Alcohols also serve as the basic materials from which esters and ketones may be obtained.

It looks highly promising to bring about reaction between, say, ethylene and water to combine them directly as alcohol. However, this work, which has been carried on intensively in England, particularly by the Imperial Chemical Industries, and further by the Bataafsche Petroleum Maatschappij, has not appeared to indicate a promising yield. The success of the operation, if it has any future, is now held to reside in the discovery of an effective catalyst.

In experiments on direct hydration of this nature,

mixtures of olefins and steam are led over catalysts at temperatures varying from 100° to 600° C. (212° to 1112° F.), generally about 100° to 350° C. (212° to 662° F.). The ratio of steam to hydrocarbons is usually low, though in some instances a ratio as high as 50:1 is used. This ratio governs, at least to some extent, the type of product obtained, alcohols being formed primarily when the ratio is high and ethers when the proportions of olefins and steam approach one another. Pressures from 1 to about 350 atmospheres have been advocated. The catalysts employed include metals and their alloys, metallic oxides and phosphates, nonvolatile mineral acids—e. g., sulfuric or phosphoric acid—and volatile organic acids, such as acetic.

## ESTERS

The reactions of alcohols with acids, acid anhydrides, or acid chlorides are familiar methods for preparing esters. The direct combination of olefins and acids is of more interest. Direct esterification of ethylene or propylene by organic acids is apparently difficult to effect but can be accomplished by first absorbing the olefin in sulfuric acid and then treating the solution with either the organic acid or a salt of the acid. Higher olefins are much more amenable to this treatment. Butyl acetate, for example, is obtained on passing butylene, at temperatures of 50° C. (122° F.) or higher and under pressures of 100 to 180 pounds per square inch, counter-current to a stream of glacial acetic acid containing some sulfuric acid. Amylene may be esterified in a similar manner. Esters so obtained may serve as solvents for various gums and resins and in the preparation of lacquers.

## KETONES

Dehydrogenation or oxidation of secondary alcohols yields ketones. The general reaction may be written:

$$\begin{matrix} R' \\ R \end{matrix}\!\!\rangle CHOH \rightarrow \begin{matrix} R' \\ R \end{matrix}\!\!\rangle CO + H_2$$

Of this class of compounds, acetone is probably the most important industrially. It is a well-known solvent for cellulose esters, for many gums and resins, for some fats and waxes, and for acetylene, particularly for the prevention of explosions during the storage of that gas. This ketone is employed also in the manufacture of smokless powder, celluloid, and certain kinds of artificial silks, as well as in the scouring of wool. Mixed with toluene or benzine, acetone yields a solvent which has been used as a dewaxing medium for lubricating oils.

Although acetone (and other ketones) has been produced largely by fermentation processes, isopropyl alcohol offers a convenient source. Dehydration of this alcohol is effected at temperatures of 250° to 430° C. in the presence of copper, metallic sulfides, or various salts of zinc, nickel, cobalt, aluminum, calcium, or magnesium. Catalysts for the vapor-phase oxidation of isopropyl alcohol include copper, brass, silver, and zinc oxide. Other metallic oxides, such as manganese dioxide and barium peroxide, have been suggested also.

Methyl ethyl ketone, made by oxidation of *sec*-butyl alcohol derived from butylene, finds application in the cellulose lacquer industry. It possesses a boiling point somewhat higher than that of acetone and because of its lower vapor pressure is less likely to cause "blushing" in lacquers. Provided this solvent could be made in quantity and sufficiently cheaply, methyl ethyl ketone would replace acetone for many purposes.

The foregoing discussion clearly shows that olefins in cracking gas can be made to unite (directly or indirectly) with water to yield alcohols, from which two main classes of derivatives are obtained, esters and ketones. Reverting again to cracking gas, the olefins present can react with chlorine to give olefin dichlorides or with hypochlorous acid to form chlorohydrins. These latter compounds in turn are converted into two other classes of derivatives—glycols and olefin oxides.

## CHLOROHYDRINS

Although known since about 1859, ethylene chlorohydrin did not become an important compound until the World War when it was used in the manufacture of mustard gas.

One method of preparing ethylene chlorohydrin consists in passing ethylene into an aqueous solution of a hypochlorite from which hypochlorus acid is set free by the addition of a weak acid, e. g., sodium hypochlorite and sodium bicarbonite or carbon dioxide. Passage of ethylene and chlorine into water or an aqueous solution of a base—e. g., sodium carbonate, either in the absence or presence of an immiscible liquid which serves as a solvent for the chlorohydrin—is another method. In such operations the temperature is kept low (about 10° to 12° C., or 50° to 53.6° F.), and the chlorohydrin concentration is maintained at about 6 to 10 percent. Reaction in the vapor phase between chlorine, ethylene, and steam, employing catalysts such as manganese dioxide, chromates, or ferric oxide, has been proposed.

The chlorohydrins of propylene and butylene have been prepared similarly. With butylene and higher olefins a considerable proportion of dichlorides is formed simultaneously which materially reduces the yields.

Chlorohydrins have been suggested as solvents for the extraction of aromatics, phenols, and sulfur and nitrogen compounds from brown-coal-tar distillates, for the preparation of wetting agents (by condensation with amines), in the making of synthetic resins

(*Continued on page 360*)

# Hydro-Metallurgical Methods *Applicable to* Oil Refining

L. C. TRESCOTT

PART II

WHILE settling is a matter which creeps into most hydro-metallurgical operations as well as into nearly any system for the chemical treatment of petroleum oils, it is not the only item which has been worked out in the mine mill which may be applied to oil refining. Many of the schemes thus developed have been applied to continuous processes. The methods can be applied to existing processes and the coming of continuous processes to the oil industry makes them that much more applicable.

In many cases the material to be settled is not a slime but a fast settling sand. Or this sand may constitute a portion of the mix which it is desired to remove before sending on the finer portions to a regular settler. In the mine mill the equipment for doing this is called a "sloughing off tank." This takes several forms, two of which are shown in Figure 1. In some cases the form is that of a narrow rectangular tank having weirs for the inlet and discharge placed at either narrow end of the tank, and in some cases provided with baffles to send the flow on a longer path. *A* in Figure 1 shows the type known as a "spitzkasten". This is also used as a classifier, separating the sands into various sizes, or it may be designed to separate the ore from the gangue. In *B* Figure 1 we have a piece of apparatus which may have a considerable application. Its use in metallurgical work is paralleled in the petroleum industry in the settling and removing of fast settling sludges from the acid treatment of gasolines, naphthas, oils in naphtha solution or any treat where the sludge drops immediately and leaves little or no pepper in the oil. In many of the continuous processes for the treatment of gasolines the sludge is settled out in a tall pipe, often against a rising current. A small conical settler has the advantage that the sludge immediately on entering the settler, falls out of the range of the flow through the tank. The flow in such a tank is in one direction. While there is a well defined movement of liquid through the tank and not a quiet displacement such as takes place in the large settlers used for slimes, there are no cross or eddy currents set up and settling conditions are greatly improved over those of a tall pipe, or of the equipment generally used for the removal of such sludges.

In the action of the various plant separators for the recovery of waste oil we have another sloughing off operation. While this is essential a separation of oil and water the weir overflow offers an ideal solution to the skimming problem. In most settlers the overflow is over the entire top of the tank but in a skimming operation this is not entirely necessary. The operation of a separator equipped with this sort of discharge is entirely automatic and requires little attention. The clean oil rises and overflows at once. The emulsions or portions which are slower to separate hang lower in the tank or reservoir and stay there until they break into oil which goes off in the overflow and water which is drawn off continuously and automatically from the bottom.

The smaller departmental separators can well be built as continuous sloughing off tanks, the oil being skimmed off at once while the water goes off through the gooseneck to the sewers and the large general separators. A settler of this sort would require little attention, would be of much smaller size and more efficient than the compartment or baffled tank type in such general use. It can be fully inclosed and as little oil is held at any time it is much safer than the batch settling type.

The general separators into which the service sewers of the refinery discharge are often large shallow reservoirs banked with earth or concrete. The effectiveness of such separators can be greatly increased by taking the oil off continuously over weirs with an overflow depth of a fraction of an inch instead of attempting to pump it off through a pipe. The skimming action of the weir will take the clear oil off as fast is at rises and will take no water with it. As a matter of fact the separation of the oil is speeded by this continuous removal. In a large separator several of these weirs may be let into the sides and the sewers should discharge well in the center of the pond, into a central feed well which extends a foot or so beneath the surface. This will assure a thorough skimming.

Where new separators are to be built, especially where space is at a premium it will be well to consider the circular type with an overflow over the entire periphery. This can be built much smaller than the pond type and will have much greater capacity per square foot of area. The water can discharge continuously through goosenecks which maintain a constant level.

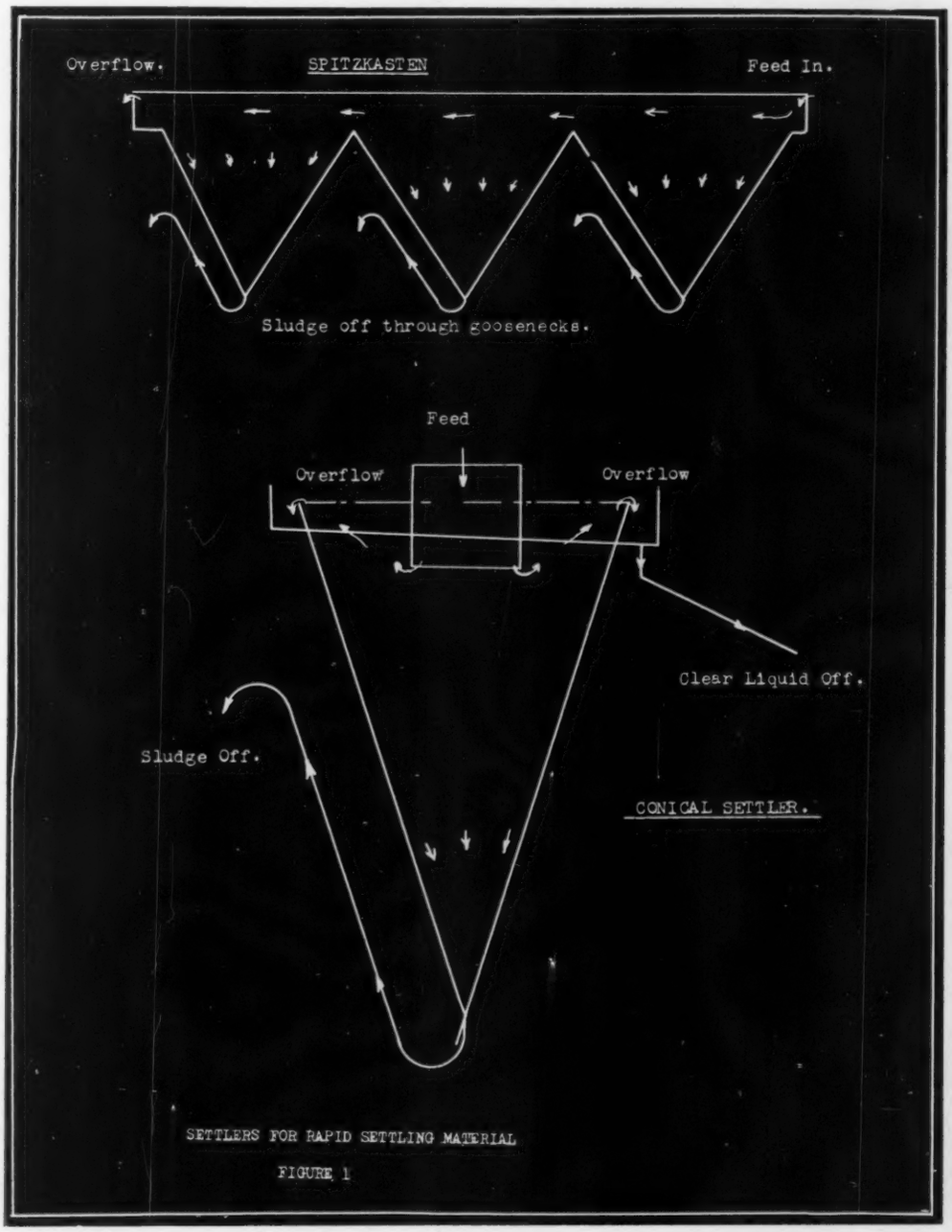
## AGITATION

Agitation in the oil industry usually receives small consideration. The general method is to drop an air line into an agitator and then "let 'er blow." Where mechanical agitation is used a paddle of some sort is put into a tank and it is speeded up to swirl the mix as violently as possible, little consideration being given to any feature of the operation other than getting the most intimate contact possible between the materials in the tank.

In metallurgical work it has long been appreciated that agitation is not a haphazard affair; that its influence reaches farther than just intimate contact, and further, that too intimate contact, too violent action, may have bad effects. It was realized that controlled agitation could be used to help in building up the particles and increasing the flocculation in the handling of slimes; that properly controlled agitation could be used to increase the leaching effect of a solution. It was recognized that the emulsifying of a mix with oil, or coating the ore particles with air in a flotation process required a different type of agitation than a leaching operation where the main point was that of keeping the solid particles in suspension. In fact it was recognized that agitation was a tool which should be used with a definite end in view.

In the discussion of settlement there was stressed the importance of promoting the flocculation and building up the particles of slimes and sludges and of pulling together soaps and suspensions to a point where they would settle. This promotion should begin in the agitator. It has long been the practice in metallurgical work to control agitation to produce specific effects in the aid of flocculation and settling. If fine particles be circulated at a rate which is slow enough they tend to pull together. Care must be taken that the agitation is not so violent as to tear down the particles that have built up. For this purpose the agitation is so planned that the particles are picked up from the bottom, carried to the surface and allowed to settle through the mix. This is a most effective way of promoting flocculation and the principal can be used to advantage in acid treating, in neutralizing and washing oils, in the separation of waxes and in other oil refining operations.

The first controlled agitation problem which is met in the oil business is that which comes into the batch treatment of lubricating oils. Here the scheme almost invariably is to run an air line to the bottom of the agitator cone and blow as violently as possible. This naturally requires large volumes of air. There is nothing positive in the circulation by such a method and there is every reason to believe that with the large volume of air in the mix, the finer particles of sludge may be coated with air which holds it in suspension. In acid treating it is often highly important to get the




SETTLERS FOR RAPID SETTLING MATERIAL

FIGURE 1

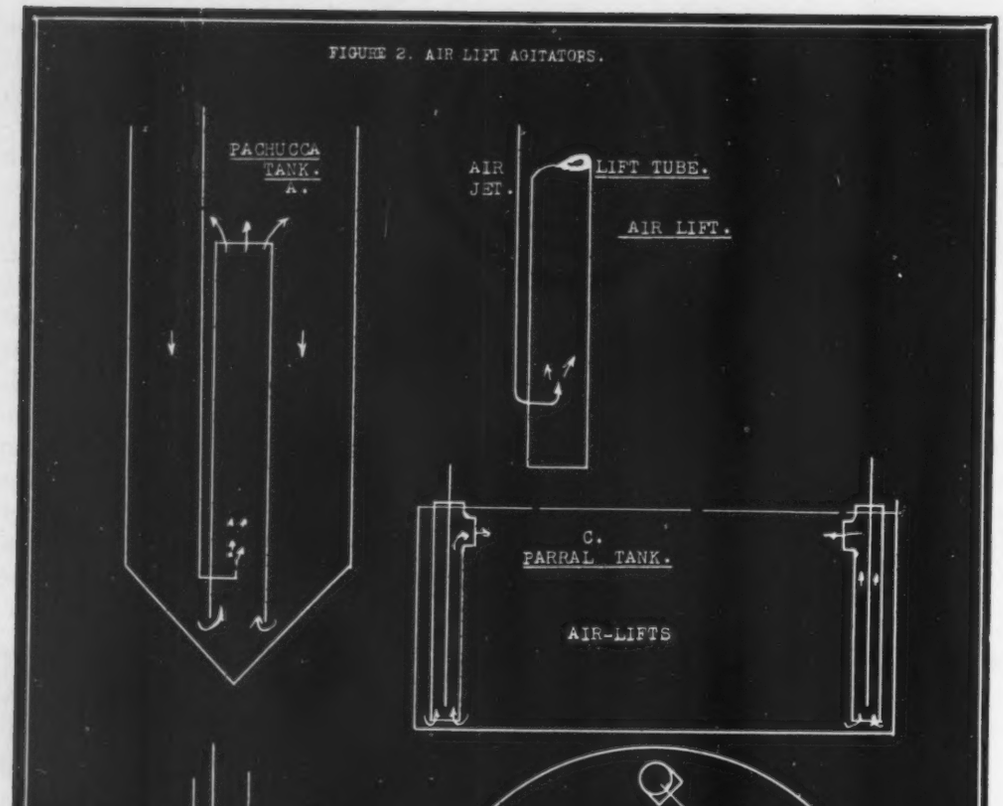


FIGURE 2. AIR LIFT AGITATORS.

sludge out of contact with the oil as soon as possible and anything that retards the settlement of the sludge is certain to effect the quality of the oil.

In the metallurgical field there was developed an agitator to meet almost the same conditions found in a large batch agitator in oil refining. This was worked out not only to give better control of agitation but also to effect a saving of compressed air. Agitation is after all the circulation of the material in the tank and an effective and economical way to do this is by use of an air lift. The Pachucca tank is a tall coned bottom tank with an air lift dropped to the bottom of the cone. This is shown in A, Figure 2. The air lift tube itself is shown in B of Figure 2. It is simply a large pipe at the bottom of which a small air jet discharges. The mixture of air and liquid inside the tube being lighter than the liquid alone which is outside, generates a verticle flow through the tube. The lift may be designed to give any desired flow and there is considerable data available for this purpose. It is a husky, simple piece of equipment, easy to install and ideally suited to operation in a tall cone bottomed agitator. The circulation it induces is positive and being such the distribution of the acid in the mix is much more complete than is brought about by the use of an uncontrolled current of air blowing through the entire agitator. Much less air is required to operate the lift than to carry on the operation without it. In most cases a three quarter-inch to one-inch pipe will carry the air to operate a large agitator.

The lift tube may be carried almost to the top of the liquid but it is better to cut it off several feet down. This gives the acid or slime a better chance to spread than if the lift be discharged near or above the surface of the liquid in the agitator.

With this type of agitator the circulation of the acid is greatly speeded. The acid being heavy will go down the full length of the agitator on the outside of the lift tube at a faster rate than the oil circulating in that space. At the bottom of the cone it is at once picked up and put in circulation again.

In the acid treatment of petroleum oils there is a tendency for the acid sludge to break up into a fine suspension which stays in the oil. With the air lift the circulation of the acid and the violence of the agitation can be so controlled that the sludge, instead of being put in suspension will be flocculated and pulled together with a minimum quantity of water.

Another type of air lift agitator is the Parral tank. This agitator was developed for the agitation of broad, low tanks as opposed to the tall high tanks of the Pachucca type. It is shown in plan and elevation in Figure 2, C. In this agitator four air lifts generally are used. They are large diameter pipes, clear at the bottom so that the contents of the tank may get into them and topped with a tee. In many cases it is possible to

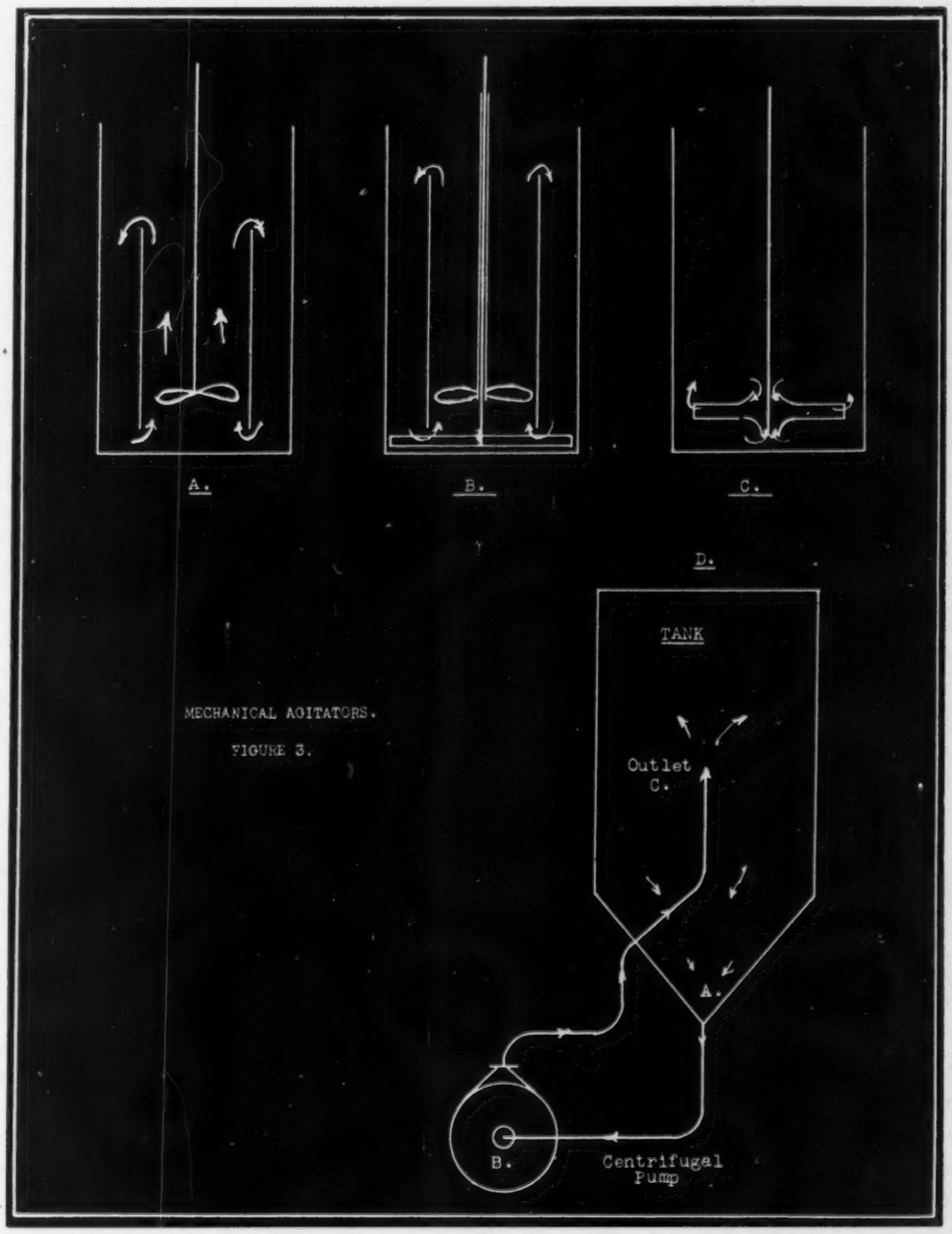
use ordinary sewer pipe built up to the desired height. The tee discharges at the side and the lifts are placed in the tank as shown with the discharges all pointed in the same direction. This not only gives a positive circulation through the tank but induces a slow swirl in one direction which increase the distribution of the suspended matter and retards slightly its settlement to the bottom of the tank again, thus providing contact without too violent agitation. As in the Pachucca tank the circulation of the heavier, faster settling particles is increased. Such action has value in leaching processes as well as in acid treating. The increase of circulation of the faster settling particles also goes far in improving the settling qualities of the batch as a whole, and is well suited to the agitation of slimes before sending them to a settler. The circulation of the heavier particles, the hindered settling due to the slow swirl, all tend toward flocculation and the increase of particle size.

In the oil business the Parral tank is well suited to neutralizing and washing operations. It provides thorough agitation under absolute control, under conditions which promote the pulling together of the soaps. It permits the operation to be carried out in a broad shallow tank, which when the agitation is stopped gives greatly improved settling conditions over the tall narrow agitators generally used.

There is a third type of air lift agitator which has worked well in metallurgical processes. This is shown in Figure 2, D. Here we have the central air lift for producing the circulation. The tank is flat bottomed and the bottom of the air lift tube is provided with rakes for pulling the settled material to the center so that it may be put in circulation again. At the top of the lift tube are long launders or troughs which extend out from the center to the sides of the tank. These are provided with openings at regular intervals. The lift tube, rakes and launders revolve slowly as a unit. The rakes pull the material to the lift and the lift discharges into the launders which distribute the material over the entire surface of the tank. It will be seen that this agitator gives a close control of the circulation as well as assisting flocculation.

When slimes and sludges which do not flow readily are used in the above agitator the rakes on the bottom of the lift tube are a decided advantage, but with free flowing sludges or soaps the rakes may be omitted.

It will be seen that the Pachucca tank and the Parral tank each has a specific application in treating oils; the tall Pachucca tank to acid treating and the lower, broader Parral tank to neutralizing and washing operations. The third type agitator may be fitted to either type tank. Where a cone bottom is used for the third type agitator, the rakes may be omitted and the lift tube dropped to the bottom of the cone.




MECHANICAL AGITATORS.

FIGURE 3.

Any of the three types of air lift agitators may be applied to continuous operation, but it will be found in the petroleum industry that they can be used best on batch processes. They can be placed easily in equipment already at hand.

So far as the continuous treatment and neutralization of petroleum oils is concerned, it will generally be found that the smaller mechanical agitator will be better suited to the job than the large air lift type. The action and conditions necessary in an air lift agitator are unchanged in a mechanical agitator. In acid treating we must first have contact between the acid and the oil. Second: the contact must not be so intimate or so violent as to put the acid sludge in suspension in the oil. The agitation then must give a speedy circulation of the acid and at the same time give the acid and the flocculated sludge an opportunity to pick up the pepper and carry it out of the oil.

This condition is not hard to realize. It means that one must experiment with agitator speeds until the best conditions are reached. It means that a type of agitator must be selected which will reduce the mechanical variables to this one matter of speed. This means the choice of an agitator which will give a circulating flow in which the acid and the oil are mixed with reasonable thoroughness rather than an agitator that tends to churn the mix, or which by sudden reversals of direction literally beats the sludge into fine suspensions.

Some mechanical agitator types which give the proper control for acid treating are shown in Figure 3. A is the conventional draft tube and propellor type. This operates on the same general principal as the air-lift except that in place of an air jet a propellor is used to induce the flow. In a small agitator which is used for acid treating it makes little difference whether the flow be up or down through the draft tube. For acid treating possibly up is best. For slimes and materials which tend to pile up and block the bottom, a down flow will keep this clear.

The draft tube may be of any desired length. For acid treating it should run to about a foot from the surface of the oil being treated. Where several agitators are being operated in series a high draft tube may be used to prevent short-circuiting.

At B, Figure 3 is shown an agitator with a draft tube, but, driven by a quill running through the hollow propellor shaft is a second slow-moving bar agitator which serves to keep the bottom clear. This type is useful in working with the clay treatment of oils, or in any case where the circulation cannot be fast enough to keep everything open.

The agitator at C, Figure 3 is built like the runner of a centrifugal pump and has been used successfully on continuous acid treating operations.

Another type of agitator which has been highly successful in oil refining is shown at D, Figure 3. This can be used to agitate any size of tank, and by using several the action of the Parral tank can be duplicated in places where it is not desirable to use air. The tank shown is built with a conical bottom. The flow is down, through the outlet A, through the centrifugal pump, B, and the outlet C, which discharges two or three feet below the surface of the liquid. This type of agitator gives excellent mixing, a positive flow, and full control. In some cases where centrifugal pump circulation is used the discharge takes place at the top or above the top of the liquid in the tank. This is a mistake, unless, as is done with the air-lift agitator, revolving arms or launders are used to distribute the flow. Where a pipe simply comes in and discharges, the circulation will be localized in only a small portion of the tank. If the discharge takes place several feet under the surface the liquid above tends to deflect the stream and spread it. Further the velocity of the discharge spends itself in useful work in agitating the mix.

A centrifugal pump used on this work must be built for rough service and the suction line from A to the pump should be as large as can be fitted to the pump.

All that has been said of agitation and settling in acid treating will apply to the problems arising in the various methods of treating oils with solvents. Here the sludges are apt to be light but the fact that the treating is done in light solvent solution helps the separation. In any system, however, it will be necessary to use controlled agitation, and it is entirely possible that the methods of continuous settling so well developed in mine mills can be applied to this proposition.

During the past few years the matter of the separation of waxes from oils has gained added prominence. Until recently the method of filter-pressing crystallized waxes from chilled solution and centrifuging the petrolatums from residual stocks was satisfactory, but at this time conditions that have arisen have turned the oil man's eyes to other methods of wax removal.

It has been found that the so-called amorphous waxes are really fine crystals which under proper conditions may be brought out and filtered. Several methods have been proposed for doing this but none of them apparently have given proper attention to building up the particles before filtration or to a proper thickening of the waxes to be filtered. If thickening works so well in the case of fine slimes there is no good reason why it will not give just as satisfactory results with fine waxes. If the procedure be properly carried out, the filter-aid which is probably necessary, can be used to help the flocculation of the waxes by mixing it in an agitator where the action is properly controlled, then thickening the batch in a continuous settler. In this way it will be found that instead of putting the entire mix through the primary filters of the system it will be possible to use them to handle a thickened slurry, while the bulk of the solution may be taken off the top of the thickener fairly clear and strained through a blotter press. In the mine mill the first move would

be to build such material up to a point it will form the best possible filter cake. There is no good reason why this should not be done in the petroleum industry.

Filtration in metallurgical work has been highly developed and has been closely tied in with agitation and settling. Where large quantities of material of little value are to be handled in order to take out a small quantity of valuable metal, naturally the cost of handling must be cut to a minimum and the efficiency of the processes used must be high.

The "sock" type of thickener is mechanically a filter. Its application to both the oil industry and metallurgical work is somewhat limited but there are places where it can be used to considerable advantage. It is essentially a low-pressure filter where a cake is picked up, pulled together, and then discharged back into the solution again to settle to the bottom of the thickener tank. This slurry is well flocculated and as it is taken off the thickener can be handled on any standard type of filter. This thickener is designed to handle slime which would either form an impenetrable cake at ordinary filter pressures or would be squeezed through the filter cloths. By working at lower pressures than would be possible if a dry cake were to be produced, it is possible to build up a well flocculated slurry.

In handling waxes it would seem that this principle could be put to use. It would mean that the bulk of the filtration could be handled at low pressure while a small regular filter operation would take care of the thickened cake. A description of this type of thickener with other applications to oil refining appears in REFINER, Vol. 12, No. 12, Dec., 1933, p. 484 and REFINER, Vol. 13, No. 4, April, 1934, p. 136.

The continuous rotary vacuum filter and the pressure leaf filter of the Sweetland type were developed in the mine mill to handle cheaply large quantities of filter cake. The application of the first type to the filtration of lubricating oils followed the same principals as its application to metallurgical work with the same satisfactory results. As in the mineral filter cake a thin cake from a continuous vacuum filter is much more dry, carries much less of the liquid being filtered than the thick cake from a higher pressure filter. The flow rates were high and the costs low. This application is treated fully in REFINER, Vol. 12, No. 10, October, 1933, p. 396.

Its application to dewaxing problems has been shown in REFINER, Vol. 12, No. 6, June, 1933, p. 238.

Its application to handling the slimes from oil refining follows the general established proceedure for such products.

The pressure leaf filter, both with stationary and rotary leaves has found a broad application in oil refining, in contact treating processes, wax filtration, and other processes. The leaf filter has many advantages over the plate and frame press, particularly where discharging the cake and putting the press back into operation is concerned. With the leaf filter this time lost from actual filtration is cut to a minimum. Labor costs are also lower as is the wear and tear on the filters and on the filter cloths.

In some cases it is possible to sluice the spent cake from the filters without opening the shells. This is common practice in metallurgical work and has been adapted to the contact process as is shown in REFINER, Vol. 13, No. 2, Feb., 1934, p. 54.

In contact treating there is little chance and perhaps little reason to do much about flocculating the cake before it goes to the filters. As it happens, the action of the clay in picking up tarry matter tends toward the formation of a granular cake from the fines and unless water be present contact filter cakes will be fairly open and will give reasonably good flow rates.

In the filtration of slimes and slurries such as are met in oil refining real attention should be given to the thickening of the slurries before any attempt is made to filter them. This will result in better filter flow rates and clarification than can be attained without the effort and in many cases slimes can be easily filtered which would not build a cake at all without preliminary flocculation.

In filtering waxes it should be possible to handle them efficiently on filters which have been developed and are standard in the metallurgical field. Here again some effort must be made to build up the wax particles, either alone or with a filter-aid to the point where they will give an reasonably open cake on a filter.

Throughout this discussion the interdependence of agitation, settling and filtration have been stressed, even harped upon. It is a matter that has been well developed in the handling of ore slimes and there is a wealth of information available, on equipment, methods, and successful work. Back numbers of the mining and matallurgical magazines will yield any number of tricks that have given good results, and it is well not to fear going back too far. The earlier stages of a development often show points which are hidden in the more complete work.

Slimes and sediments cannot usually be handled by formulas, but with the data at hand and a reasonable application of common sense it should be possible to bring much of the success of the mine mill along this line to the petroleum industry.

# *Elements of* . . . Stabilization

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## PART III

**IN previous articles in this series the writer has discussed various phases of high pressure stabilization. In further extension and expansion of the subject he describes in the accompanying discussion methods whereby maximum yields of substantially pure hydrocarbon fractions may be produced from natural gas, including separation of methane, ethane, propane, isobutane and butane and the pentanes.**

REGARDLESS of the purpose for which the stabilizing column may be used an accurate and sensitive control system is vitally essential to maximum yields of high quality product. A system that would give the greatest coordination between temperature, pressure and flow would seem highly desirable as these are interdependent conditions. Every variation in pressure assumes a corresponding change in temperature for the maintenance of uniform product. Flow also enters into the picture, as temperature-pressure variations influence the tower capacity and its functional characteristics. Liquid levels are dependent upon flow. Insofar as flow is concerned that method of control which will give the most immediate action with the minimum fluctuation would appear desirable. In other words there should be, if possible, a substantially instantaneous remedial action and that should be neither an underdose or an overdose.

Some of the more recent developments in control instruments would appear worthy of careful consideration by natural gasoline plant engineers. For practical purposes, the experience gained with installation in oil refineries should prove of value. No other industry has had more experience in automatic control than that of oil refining.

Many raw natural gasoline stocks contain the isomers as well as the normal hydrocarbons. These hydrocarbons have boiling points intermediate between the normal homologs. For illustration, isobutane is intermediate in boiling point between propane and normal butane, as are the isopentanes intermediate between normal butane and normal pentane. To make a close separation of such constituents requires a marked accuracy of control. The difficulty of making a close separation between isobutane and butane has been one of the contributing causes of too low yields and/or poor products in many natural gasoline plants. The close separation of propane and isobutane in bottled gas manufacture has also been a problem for automatic temperature pressure and flow control.

For flow control at high pressures and to avoid the use of stuffing boxes, various devices may be used such as for example the magnetic clutch type instrument. Forged steel fittings are in general use with such installations.

### REFRIGERATION

The use of artificial refrigeration as an aid to stabilization has not been fully appreciated. As has been stated in previous articles, there is an advantage in the use of low pressures with low temperatures insofar as the separation of close fractions is concerned.

The volatility ratio, that is, the ratio of the vapor pressure of one component to that of another at corresponding temperatures, is an index to the ease of separation. At low pressures the volatility ratio becomes more pronounced and, therefore, as in analytical work, low temperatures are used. Natural gasoline plants as a rule have an excess of propane in the raw stock, or if not, it is available in the incoming gas. Propane may be used as a refrigerant. The use of propane or a gas which is substantially propane as a refrigerant introduces no additional foreign substance into the system.

It would seem that cooling by direct expansion into the cooler equipment (reflux condensers, etc.) would be most practical on account of the high coefficient of heat transfer and the preclusion of the use of brine and such equipment as is needed for its handling. For the separation of propane from a raw stock, propane may itself be used to cool by direct

expansion and contact. That is, the overhead gases from the column could be compressed and condensed so as to yield a liquid essentially propane which could be used for direct reflux. This would permit a lower operating pressure on the column, the compressor taking the place of a pump for the necessary elevation of pressure above that of the column operating pressure. In fact compression of the overhead gases may be used for the separation of isobutane and butane from the raw stock in the manner indicated for the separation of propane. This would make the column operating pressure independent of the pressure obtainable by means of the ordinary plant cooling medium, which as a rule is water. The general operating technique therefore becomes more flexible as a wider range of operating pressure is available. By the use of heated suction lines to the compressor and operating at a vacuum the pentanes also could be separated using a low pressure column. Here, however, great care would have to be exercised in order to preclude the possibility of air infiltration into the system. This would be a source of danger as to explosion and also detrimental to the operating efficiency.

The factors to be taken into account in the evaluation of a refrigerant aside from that of specific practicability as a by-product are as follows:

(1) Chemical formula.
(2) Molecular weight.
(3) Normal boiling point.
(4) Melting point.
(5) Critical temperature.
(6) Critical pressure.
(7) Density of liquid at 32°F. (Water = 1).
(8) Density of gas, pound per cubic foot, 32°F., 1 atm.
(9) Density of gas at 32°F. and 1 atm. (air = 1).
(10) Specific heat at constant pressure.
(11) Specific heat at constant volume.
(12) Ratio of specific heat at constant pressure to specific heat at constant volume = $Cp/C_v = \propto$.
(13) Comparative volume displacement per unit of refrigeration.
(14) The vapor pressure curves.
(15) The volume of liquid in cubic feet per pound under various operating conditions (curve).
(16) The volume of vapor under various operating conditions (curve).
(17) The sensible heat of the liquid B.t.u. per pound.
(18) The sensible heat of the vapor B.t.u. per pound.
(19) The latent heat of vaporization B.t.u. per pound.

Most of this information for the lower hydrocarbons has been given by the writer in past articles. Tables of the thermodynamic properties are now in the course of preparation similar to those used for ammonia and steam.

## LOW PRESSURE SYSTEM

Future or even present conditions within the natural gasoline industry might warrant consideration for some practical means of the *in toto* separation of the components of natural gas into substantially pure fractions. From an engineering standpoint such a system is possible and practicable, the question being whether or not such a system would be practical from the industrial view point under present conditions.

For chemical processing it is, as a rule, of advantage to have substantially pure raw materials. Insofar as natural gas is concerned, the raw materials available are the components i.e. methane, ethane, propane, isobutane, butane, isopentane, pentane, the hexanes, etc., up the line of homologs.

Methane is the most commonly occurring and most abundant of the hydrocarbon gases so the mere separation of this material in order to have a supply in the nearly pure state would not be of much advantage. However, this may be done by applying the principles already worked out for the liquefaction and separation of air into its constituents, should the occasion demand.

On the other hand for practical purposes the *in toto* recovery of ethane from a raw gas condensate would necessitate the condensation of the overhead vapor, methane, for the use as reflux. For such purposes several modes of operation may be suggested, as for illustration, a cut could be made between the methane and ethane in the raw gasoline stock from the first column or a gross cut could be made between any of the higher constituents. Practically, and without going into a detailed analysis as to the reasons, it would seem preferable to make a gross cut between the butanes and the pentanes in the first column and then use the overhead condensate as a secondary raw stock for further processing. In such event the bottom product from Column No. 1 could be used too as a secondary stock, or it could be used possibly as a high grade high gravity motor or aviation fuel, or else blended with butane to give the maximum yield of required specification natural gasoline. The markets for such products would determine the operating procedure to be used for the bottom product of the first column.

Let us say for illustrative purposes that a gross cut has been made between the butanes and the pentanes from the raw stock and that there is available two secondary raw stocks for further processing i.e. bottoms and overhead condensate. Our attention will first be directed toward the separation of the overhead condensate into its constituents. (Note: A compressor will in all probability have to be used to effect total condensation). This condensate will pass from the accumulators through the necessary heaters, heat exchangers and through regulatory equipment to the column. This column should be highly insulated as it will operate at low temperatures, and the entire system will be self refrigerating after operations are started.

All of the overhead vapors from the column will pass to a compressor at first until the column is lowered to its operating temperature by internal

vaporization of the feed and reflux condensate. Gradually the system will begin to cool until the plates are covered with liquid and the vapors passing overhead are substantially methane. Methane of itself will not condense at any pressure within the region of practical temperatures for the cooling medium. In order to affect its condensation an expander will have to work in conjunction with the compression and water cooling system. The power generated by expansion may be dissipated or utilized in the form of heat through a generator and rheostat. The generator and rheostat will act as a balance wheel for the system.

The discharge from the expander will consist of both liquid and gaseous methane, but this need occasion no worry as there will in all probability be an excess of methane over that required for reflux. The condensate which will also be methane will be returned to the column in amounts such as is required for the reflux essential to efficient fractionation. It goes without saying that all cold lines and other cold equipment should be very thoroughly insulated to stop heat infiltration which would increase the work and size of the system. The fractionator for the separation of methane would have to be designed for high pressures and low temperatures, but otherwise it would be very simple and have relatively few plates (not over 10) as the boiling points of ethane and methane are so far apart at any point of corresponding pressures. In all low temperature systems of this type it is imperative that the materials under processing must be free of all water vapor as the gradual accumulation of ice would hinder its functioning. Alumina or silica gel may be used for this purpose in a manner similar to that used for the preparation of anhydrous c. p. ethylene such as is used for medicinal purposes.

The best place to effect dehydration would be at some point in the system just previous to the feed plate of the column proper. Refrigeration and compression are both of advantage for the removal of the major portion of the moisture.

## SEPARATION OF ETHANE

The separation of ethane constitutes neither a serious problem in fractionation or condensation. An outline of a general procedure for operation might be as follows:

The de-methanized bottom product from the column just discussed would pass by its own pressure to the ethane column. In this column a larger number of plates would be required as the boiling points of ethane and propane are closer together than those of methane and ethane. At atmospheric pressure ethane boils at — 126.9°F. and propane at — 48.1 and at their respective critical temperatures 89.8°F. and 204.1°F. The critical pressure of ethane is 718 pounds per square inch absolute. As no fractionation can take place at the critical point some operating pressure lower than 718 pounds must be used with some reflux condenser temperature lower than 89.8°F. The pressures and temperatures are interdependent so that the lowest practicable temperature will determine the operating pressure. If so desired, and if sufficient, the cold gaseous methane from the expander accumulator of the first column could be used to supplement the cooling obtainable from plant water to provide reflux. In the event that this procedure might be decided upon, no compressor and expander system such as was discussed for the separation of methane would be required. This might prove of economic advantage. However, the same might not prove as practical from an operating standpoint.

In case the ethane is to be obtained in a manner similar to that described for methane, the equipment used would be much smaller and the power requirements much less. On the whole, however, it might seem advisable to condense the ethane necessary for use and for reflux by supplementing the cooling with water some means of artificial refrigeration or else allow a small amount to go to waste from an expansion system. Naturally the excess liquid ethane over that required for reflux would be passed from the accumulator to the point of use or storage through liquid level control. Ethane at the present time shows promise of becoming a valuable raw material for chemical processing. It is more chemically reactive than methane.

In case the gaseous methane from the expander accumulator is used for the condensation of ethane as reflux for the ethane column subsequent to water cooling a high degree of heat transfer efficiency should be possible. For example, the temperature of the equilibrium mixture of liquid methane and vapor is — 148.0°F. at 363 pounds gauge. At atmospheric pressure it is — 298°F. so it is easily seen that the wide temperature difference conducive to good heat exchange is available. If a temperature of 32°F. is maintained on the ethane column the operating pressure would be about 332 pounds gauge.

At 32°F. the vapor pressure of propane is 73.5 pounds absolute so the volatility ratio of ethane to propane is $\frac{73.54}{346.20} = \alpha = 0.213$. This is indicative of the ease of separation at at $\alpha = 1$. No separation is possible. Twelve plates of average efficiency should suffice for the separation of ethane.

## SEPARATION OF PROPANE

As we go higher into the series of homologous hydrocarbons the requirements for artificial refrigeration disappear, although such may be advisable for flexibility of operation. Propane is at present some-

what extensively used for bottled fuel gas and to a lesser extent as a raw material for chemical processing. Established methods are already in use for its separation. As one of the intermediate hydrocarbons between gas and liquid and because of its response to heat and chemical treatment, greater interest should be shown in methods for its recovery. Falling next higher in the series of homologs above ethane it is one of the more abundant components of natural gas. It would be reasonably safe to state that propane constitutes a greater proportion by analysis of the total natural gas produced than of the total higher constituents, including the components of natural gasoline. If means of profitable outlets are obtainable, there is a possibility that the value of propane in natural gas may be in excess of that of the natural gasoline.

The critical temperature of propane is 204.1°F. and the critical pressure 662 pounds absolute. It is, therefore, entirely practical to operate a column at superatmospheric pressures for its separation without the use of other than ordinary plant cooling water through overhead (reflux) condenser. The mixture from the ethane column will consist of a mixture of propane, isobutane and butane. The problem of fractionation becomes somewhat more involved insofar as the column proper is concerned, but the fractionation system as a whole becomes simplified as it is in the realm of ordinary operating pressures and temperatures. A column for the separation of propane may be either operated at low temperatures and low pressures using a compressor with condensation after compression or else the column may operate under autogenerated pressure and the necessary reflux either returned by straight overhead condensers or by pumping.

The difference in boiling points between propane and isobutane however is not nearly so great as that between propane and ethane or between ethane and methane.

At atmospheric pressure, propane boils at — 48.1° F. and isobutane at about 14°F. At 86°F. the volatility ratio is $\frac{59.5}{157.7} = \alpha = 0.38$, so there is nothing to indicate other than that an extremely close separation may be accomplished. For average conditions of condenser cooling water without the use of compression before condensation of the overhead vapors, an operating pressure not in excess of 175 pounds on the column should be sufficient. As in the case of ethane, the excess propane above the requirements would be drawn off through liquid level control from the accumulator tank.

## SEPARATION OF ISOBUTANE AND BUTANE

The residue (bottoms from the propane column will consist of isobutane and butane. The difference in the physical and chemical properties of these two constituents are of structure rather than formula. In other words all of the hydrocarbons are made up of carbon and hydrogen atoms as the name suggests. Butane and isobutane both have the same number of hydrogen and carbon atoms; there are four carbons and 10 hydrogens but the structure is different just the same as various shaped structures may be made with two shapes of bricks. This molecular structure influences the chemical and physical properties.

However, there is a closeness of relationship between the two and for this reason many of the physical properties are close together. For illustration, the boiling points of isobutane and butane at atmospheric pressure are 13.6°F. and 33.1°F. respectively. The volatility ratio between the two is, therefore, less pronounced and the difficulty of separation by fractionation is greater. At 86°F. the volatility ratio is $\frac{41.6}{60.5} = \alpha = 0.688$. For the separation of isobutane from butane a greater number of plates are required than for any of the previous separations discussed, and it is imperative that sensitive control instruments be used for quick temperature, pressure and flow response. A 40-plate column operating at 50 pounds gauge should be sufficient for close separation although calculations would indicate much fewer plates would be required. Condensation of the overhead isobutane vapors from the column may be accomplished either after compression or without compression under column pressure. There should be little difficulty in either case. The butane bottoms from the column may be used to swell the volume of the product of the primary stabilizer product so as to give a maximum yield within specifications. There are also limited markets for both isobutane and butane and both may be used either independently or together to bring up the B.t.u. value of bottled gas. These constituents also have great possibilities for utilization in chemical processing such as cracking or other treatment for the production of motor fuel.

## SEPARATION OF PENTANES

If it is desired to separate the three individual pentanes from each other in the substantially pure state, an overhead cut should be made in a column auxiliary to the primary stabilizer. This may be done at atmospheric or slightly super atmospheric pressure. The fraction containing the pentanes would then be sent to one or two columns in series depending upon whether there were two or three pentanes present. Fractionation would of the pentanes take place at substantially atmospheric pressure. A great number of plates would be required as the boiling points of pentane and its isomers are close together.

# Change *of Absorption Oils* Secures Higher Yields

WESTOAK Gasoline Company, operating a well-pressure absorption natural gasoline plant near Sayre, Beckham County, Western Oklahoma, is running with an efficiency of 1.31 against Podbielniak analysis on the raw gas before it enters the absorption column. Analysis of this gas indicates the following:

| | Mol Percent | G.P.M. |
|---|---|---|
| Methane | 95.69 | |
| Ethane | 1.98 | |
| Propane | 1.03 | 0.28 |
| Iso-butane | 0.34 | 0.11 |
| N-butane | 0.41 | 0.13 |
| Pentane and heavier | 0.55 | 0.20 |
| | 100.00 | 0.72 |

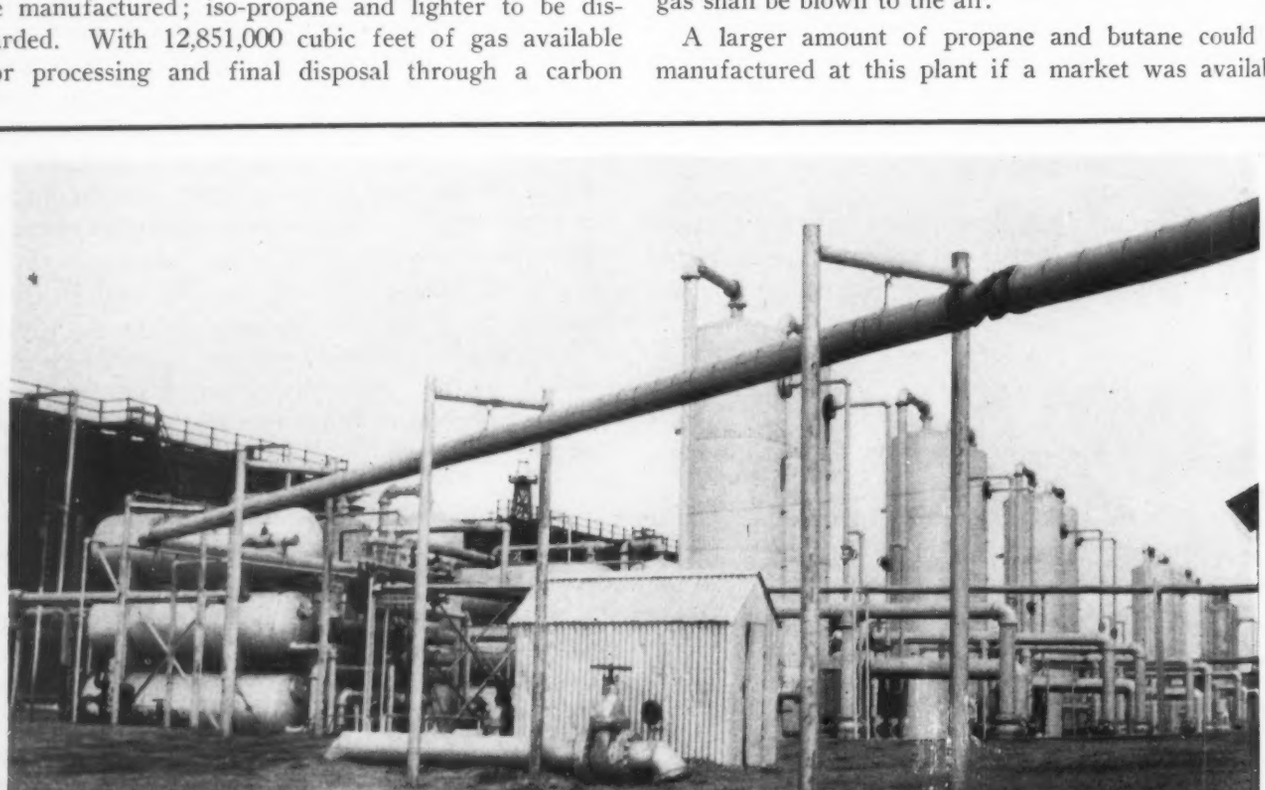
The estimate indicated that a well operated plant could reasonably expect to extract 0.33 gallons per thousand cubic feet, basing the calculations upon presumption that a product approximating 26/70 was to be manufactured; iso-propane and lighter to be discarded. With 12,851,000 cubic feet of gas available for processing and final disposal through a carbon black plant, there should be only 4240 gallons of 26/70 gasoline, should all of it be extracted. Actual recovery through the plant shows a production of 7586 gallons from the above volume of gas; the recovered product being divided as follows:

> 26/70 gasoline, 89.8 A.P.I., 5586 gallons; commercially pure butane, 1700 gallons; commercially pure propane, 300 gallons.

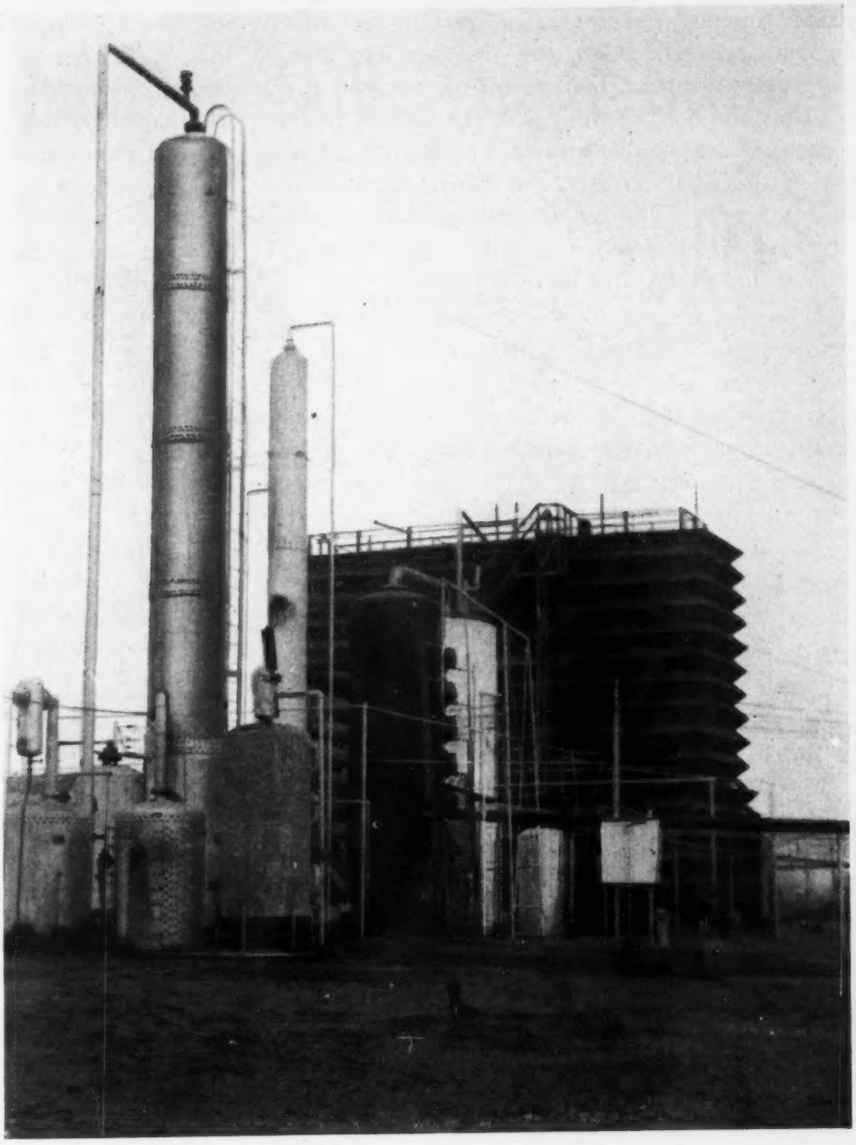
Showing that with a recovery of 5586 gallons of 26/70 gasoline, the plant factor is 1.31 with a recovery of 0.434 gallons per thousand cubic feet of gas processed.

The production of any hydrocarbon fraction, or combination of fractions, is limited to the amount of stripped dry gas residue that can be processed through a carbon black plant. Only while this plant is being operated can any volume of gas be passed through the natural gasoline recovery system because of an agreement among producers and consumers of gas that no gas shall be blown to the air.

A larger amount of propane and butane could be manufactured at this plant if a market was available



***Battery of six units of absorbers in Texas Panhandle which has recently converted to heavy oil.***



*Absorption and distillation equipment, Westoak Gasoline Company, Sayre, Oklahoma.*

for these products. In that section of the country, only the gallonage of propane and butane can be successfully handled that can be readily be sold; and sales for these fractions depend upon local conditions altogether. Some of the smaller towns in Western Oklahoma do not have natural gas available for domestic consumption, and a comparatively few have made arrangements for the utilization of butane. Propane is marketed in the same manner and under the same handicap because so few consumers of fuel have converted their stoves so that they can burn "bottled" gas in their homes.

Butane has been used successfully in drilling one of the company's wells, isolated from gas fuel lines, and the butane that is used for this purpose was trucked to the location and revaporized at the well for boiler fuel. This well was completed recently, drilled from spudding in to bottom hole with butane as the only fuel for generating steam in the boilers. Consequently, the production of this cut from the stabilizers is limited to the outlet, and since the well has been finished, only that amount needed for service in the towns that have carbureting facilities for converting the liquid butane into domestic fuel is manufactured.

Gas is produced in the Sayer field adjacent to the gasoline plant and brought into the plant under a pressure of 300 pounds gauge, and carries a temperature sufficiently low that it is not necessary to cool it before it is introduced into the single absorber column. When this plant was placed in operation, it was the belief among natural gasoline manufacturers generally that a relatively low boiling absorption oil was better suited for the recovery of the desirable hydrocarbons carried with the gas than one with an initial boiling point considerably higher than the end point of the product that was to be manufactured. The reason for this belief was that many laboratory experiments had been made, using oils of different boiling ranges, and these tests showed that more gasoline could be extracted from a given amount of gas if the absorbing oil had an initial boiling point fairly close to the end point of the natural gasoline, than if one was used that had a high initial boiling point, and whose characteristics were remotely removed from those of the natural.

Working upon the accepted ideas concerning absorption oils, Westoak Gasoline Company started its plant with an absorption oil that had a relative low initial and a short boiling range, and operated for some time in that manner. But after several months of comparatively smooth operation it was discovered that the amount of gasoline recovered from the gas did not check with the analytical column analysis, notwithstanding that the residue was dry and did not carry an

appreciable amount of recoverable fractions as shown with a charcoal test. Also, the absorber had recurrent tendencies to prime, holding up the absorption oil on the plates and not returning it to the base as rapidly as desirable. Considerable saturated absorption oil was trapped in the afterscrubber and blown back to the system frequently. The mains leading to the carbon black became contaminated with this oil so that it was necessary to install drips in the low places.

In making an attempt to remedy the situation and recover more gasoline, a heavier oil was introduced into the plant system after thoroughly draining all of the lighter oil that had been previously used. Immediately production of gasoline was raised without any appreciable change in the character of the gas being processed, or any change in the operating conditions in the absorber or distillation unit. Since that time, only an oil having the following general characteristics has been used at this plant as an absorbing medium.

Distillation:

| | |
|---|---|
| Initial boiling point | 482 °F. |
| 10 percent over at | 518 °F. |
| 20 percent over at | 530 °F. |
| 30 percent over at | 536 °F. |
| 40 percent over at | 544 °F. |
| 50 percent over at | 550 °F. |
| 60 percent over at | 559 °F. |
| 70 percent over at | 568 °F. |
| 80 percent over at | 579 °F. |
| 90 percent over at | 594 °F. |
| Maximum | 630 °F. |
| Recovery, percent | 97.0 |
| Residue, percent | 1.0 |
| Loss in distillation, percent | 2.0 |
| Initial to 20 percent over range | 48 °F. |
| Over at 500 °F., percent | 1.5 |
| Over at 550 °F., percent | 50.0 |
| Over at 600 °F., percent | 92.5 |
| Distillation range | 148 °F. |
| Molecular weight | 217 |
| Gravity, A.P.I. | 36.7 |
| Doctor | Sweet |
| Corrosion, 3 hours at 212 °F. | Non-corrosive |
| Flash point, Cleveland open cup | 260 °F. |
| Fire point | 285 °F. |
| Heat, specific at 100 °F. | 0.477 |
| Heat, specific at 70 °F. | 0.460 |
| Heat, specific at 40 °F. | 0.440 |
| Heat, specific at 10 °F. | 0.422 |
| Heat of vaporization, B.t.u. gal | 686 |
| Heat of vaporization, B.t.u. lb. | 98 |
| Viscosity, SU, at 100 °F. | 39 |
| Viscosity, SU, 70 °F. | 46 |
| Viscosity SU, at 40 °F. | 63 |
| Weight per gallon | 7.005 |

Flow of both gas and oil through this plant is similar to that of others of like manufacture, the gas coming in at 300 pounds and absorbed in a single column that is 42 feet in height, four feet in diameter, containing 16 bubble trays with 24-inch spacing. The oil is pumped to this column through a multi-stage centrifugal pump, connected directly to an electric motor, and the rate of circulation, as of March 31, 1934, was 15 gallons per thousand cubic feet of gas. Oil to the absorber has a temperature of 82 °F. on that date, and carrying 73 °F. at the draw off control. Atmospheric conditions were: wet bulb 52° F. dry bulb 60 °F., and the coil water carried a temperature of 56 °F.

Distillation of the oil is carried out in a standard unit operated at 50 pounds and under a temperature of 350 °F. The recovered gasoline in the accumulator, or run tank had a temperature of 62 °F. and a gravity of 94° A.P.I. Vapors from the high pressure vent tank and those from the accumulator are piped to a tail absorber, contacted in this column with a split stream of oil from the main circulating pump, and recovered with the other production in the same distillation and condensing equipment. This unit being operated under a pressure much lower than the main column requires that the oil from the surge tank be pumped to the high pressure vent tank, and to accomplish this, a small motor driven centrifugal pump is used, located near the base of the column.

The raw gasoline is processed in a standard fractionator where it is stabilized to a gravity of 89.8° AP.I. while the overhead vapors, principally propane and butane, are processed in a smaller tower for separation. Propane at the rate of 300 gallons per day is recovered and bottled for distribution in the surrounding country. The butane is trapped from the after-coolers at the base of the separating fractionator, sent to storage and transported to point of consumption by tank truck. If the market for this product becomes limited, excess amount above the local requirements is vented to the dry gas lines to the carbon black plant.

Since this company has changed over to a heavier grade of absorption oil, several of the other manufacturers in different parts of the country have done likewise, and all report a greater yield of 26/70 gasoline than when operating with the light oil, and particularly when the gas that they are processing carries a low G.P.M. of recoverable fractions. One company in the Texas Panhandle, operating with identical oil, reports an absorption oil loss of only 0.09, while processing at the rate of 115,000,000 cubic feet per day, while the absorbers are overloaded almost 25 percent, and making around 43,000 gallons daily of 26/70 gasoline.

Another manufacturer having a large plant which is operated in two pressure sections, one of which contains three units working at 35 pounds, and the other having three units also, but having a back pressure applied of 75 pounds, had been using a low initial boiling point absorption oil, and after changing over to the higher initial, short boiling range material reports that a large increase has been obtained.

# Use of Propane in Lube Oil Refining

(*Continued from page* 336)

extracted oil from a typical asphaltic residuum. With a wax-bearing residuum, wax begins to precipitate from the propane-oil phase at a temperature generally between 35 and 45° F., depending largely on the wax concentration and propane ratio. As the temperature is lowered successively, additional quantities of wax are precipitated until at minus 42° F. (the boiling point of propane at atmospheric pressure) the wax is practically completely precipitated with a small amount of additional asphaltic or pseudo-asphaltic material. When dewaxing is to be accomplished, the propane and the residuum are usually at such temperatures that the resulting mixture is between 80 and 100° F., and any appreciable amount of asphaltic phase appearing is generally removed before the wax cloud point is reached upon chilling.

## DEWAXING WITH PROPANE

The use of propane was first proposed for dewaxing in the hope of materially reducing the expense of the dewaxing operation which had acted as a hindrance in utilizing waxy crudes available, and had caused the production of lubricating oils in California to be limited practically entirely to low cold-test crudes of the Gulf-Coastal type. At that time it appeared that unless a very economical dewaxing process could be devised, it would not be practical from an economic standpoint to manufacture paraffin-base lubricating oils from the stocks available. The use of propane as an internal refrigerant and wax precipitant appeared to offer a solution to this problem. During the long time of experimentation and development, a large variety of oils has been tested for dewaxing by means of liquid propane, and it has been possible to devise a successful dewaxing procedure for each sample of oil tested to date.

The ease and efficiency with which the refrigeration required for dewaxing can be accomplished by evaporating propane in the solution to be dewaxed is readily obvious; but the use of propane as the diluent at the moment of wax removal, by providing an excess of liquid propane over that required for refrigeration, provides a wax-precipitating medium of far greater efficiency than could be predicted on the basis of the wax-precipitating efficiencies of naphthas or pure hydrocarbons of different boiling ranges or average molecular weights. For example, it had been found that the solubility of wax in petroleum distillates increased as the average boiling point was decreased,[1] which indicated greater efficiency of wax removal by the use of kerosene and gas-oil fractions as diluents in dewaxing. This was also confirmed by common experience in pressing wax

***General view of Union Oil Company's propane dewaxing and deasphalting plant. Chillers in the foreground, solvent recovery system centrally located and compression plant in the right background.***

**TABLE 1**

***Properties and Yields of Oil and Asphalt Produced from Poso Creek Residuum (Topped to 66 Sec. Saybolt Universal Viscosity at 100° F. on Overhead Stream) by Extraction with Low-Molecular-Weight Hydrocarbon Solvents***

| SOLVENT | (10 Volumes of Solvent at 80 Deg. F.) Yield of Oil (Per Cent by Volume Residuum) | Yield of Asphalt (Per Cent by Volume Residuum) | Properties of Extracted Oil: Gravity Deg. A.P.I. at 60° F. | Viscosity Saybolt Universal at: 100° F. | 210° F. | Carbon Residue (Per Cent) | Color N.P.A. | Properties of Asphaltic Residue: Specific Gravity at 60° F. | Melting Point (Deg. F.) | Penetration at 77° F. | Ductility at 77° F. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Ethane | 11.0 | 89.0 | 24.2 | 255 | ... | 0.077 | 3¼ | 0.98 | ... | soft | 0 |
| Propane (boiling point,— 42 to — 40 deg. F.) | 75.0 | 25.0 | 17.5 | 2,100 | 94 | 2.35 | opaque | 1.063 | 176 | 2 | 0 |
| Propane, 50 percent Butane, 50 percent (boiling point,— 42 to + 36 deg. F.) | 87.3 | 12.7 | 15.8 | 4,000 | 119 | 4.2 | opaque | ... | 275 | 0 | 0 |
| Butane 50 percent isobutane, 50 percent normal butane (boiling point, 14 to 31 deg. F.) | 89.2 | 10.8 | 15.7 | 5,000 | 139 | 5.3 | opaque | 1.107 | 295 | 0 | 0 |
| Butane (boiling point, 30 to 36 deg. F.) | 88.8 | 11.2 | 15.2 | 4,600 | 139 | 5.12 | opaque | ... | 307 | 0 | 0 |
| Pentane (boiling point, 82 to 100 deg. F.) | 95.20 | 4.8 | 14.5 | 10,000 | 192 | 6.23 | opaque | ... | 320 | 0 | 0 |
| Hexane (boiling point, 140 to 156 deg. F.) | 98.0 | 2.0 | 13.8 | 15,000 | 208 | 7.09 | opaque | ... | 325 | 0 | 0 |

distillate, in which gas oil actually served as the diluent or wax precipitant, and in which pour points practically the same as the dewaxing temperature were obtained. With the pure compounds, *i*-decane, *n*-octane, *n*-heptane, *n*-hexane, and *n*-pentane, respectively, a consistent increase in wax solubility with decreasing molecular weight had been found[2]; and it appeared that a regular relationship existed which caused the use of low-boiling hydrocarbon diluents to be regarded with disfavor. However, actual experiments demonstrated the effectiveness of liquid propane the fraction generally used

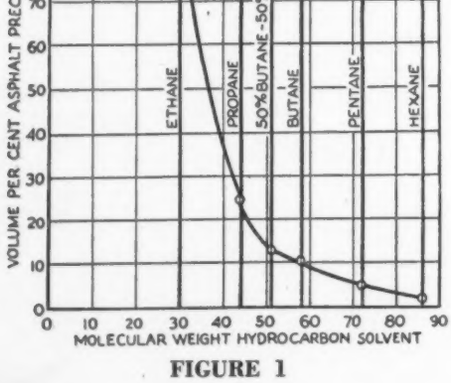



**FIGURE 1**

being natural-gasoline stabilizer overhead consisting of about 80 percent propane with the remainder divided between ethane and the butanes) as a dewaxing solvent, despite the poor behavior of the light naphthas in comparison to the heavier fractions. For comparison of results to be expected with other possible dewaxing solvents, solubility curves of paraffin wax with a melting point of 123°F. have been determined in propane and a number of other solvents, some of which are shown in Figure 6. The solubilities in propane were determined by sealing weighed quantities of wax and propane at liquid air temperature in ampules of four mm. Pyrex glass tubing, warming the ampules to room temperature, and then determining the temperature at which the first precipitation of wax occurred on cooling in a gasoline bath in an unsilvered Dewar cylinder. The temperature of the gasoline bath was controlled

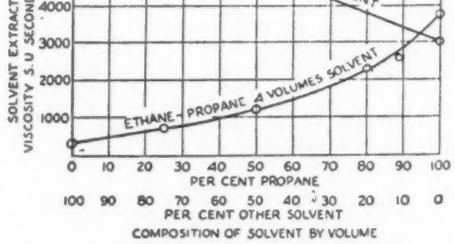

EFFECT OF PROPORTION OF BUTANES AND ETHANE IN PROPANE ON VISCOSITY OF OIL EXTRACTED FROM POSO CREEK RESIDUUM AT 80°F
SOLVENT EXTRACTED OIL VISCOSITY S.U SECONDS AT 100°F.
BUTANES-PROPANE 5 VOLUMES SOLVENT
ETHANE-PROPANE 4 VOLUMES SOLVENT
PER CENT PROPANE
PER CENT OTHER SOLVENT
COMPOSITION OF SOLVENT BY VOLUME

**FIGURE 2**

by an immersed copper coil, into which liquid air or liquid propane was introduced as needed from a Dewar flask. By changing the temperature of the bath very slowly, and holding at the cloud point, the temperatures of complete wax miscibility can be determined easily to within less than 0.5°F. The data for the other solvents were obtained in a somewhat similar manner, except that measured quantities of solvent and wax were introduced into a test tube carrying a thermometer and coiled-wire stirrer through a cork stopper. It is

realized that the wax-solubility data obtained in this manner are not necessarily comparable as regards numerical values with the results obtained on a different sample of wax by the same or a different method. The data are strictly comparable among themselves, however, and can be used to rate the different solvents according to their efficiencies as wax precipitants. To facilitate possible comparison, the physical characteristics of the sample of wax used are shown in Table 2.

**TABLE 2**

***Inspection Data on Paraffin Wax Used in Solubility Experiments***

| | |
|---|---|
| Melting point, deg. F. | 123 |
| Gravity, deg. A.P.I. at 60 deg. from test at 130° F. | 43.5 |
| Viscosity, Saybolt Universal: | |
| at 150° F. | 47 |
| at 210° F. | 39 |
| Molecular weight | 369 |
| Vacuum distillation at 1 mm. pressure: | |
| Initial boiling point | 440° F |
| 10 per cent point | 454° F. |
| 20 per cent point | 458° F. |
| 30 per cent point | 465° F. |
| 40 per cent point | 470° F. |
| 50 per cent point | 480° F. |
| 60 per cent point | 490° F. |
| 70 per cent point | 502° F |
| 80 per cent point | 515° F. |
| 90 per cent point | 532° F. |

It is seen from Figure 6 that the solubility of wax in the naphthas increases as the boiling range is lowered, but a reversal of this behavior occurs upon continuing to decrease the boiling point of the solvent; so that propane is a poorer solvent for wax, and, therefore, a more efficient wax precipitant, even than kerosene. The naphthas, the kerosene, and propane were all obtained from Santa Fe Springs crude.

Upon further examination of the wax-solubility curves in Figure 6, the very poor wax-precipitating efficiency of naphthenic solvents, illustrated by cyclohexene and methyl cyclohexane, as shown by higher wax solubilities in these solvents, is quite striking; and indicates that it is desirable to avoid the presence of at least the lower-molecular-weight naphthenes in the dewaxing medium. In other words, it would not be sufficient merely to employ low-boiling gasoline fractions, such as petroleum ethers from many crudes; but it is desirable from this standpoint to use the very low-boiling fractions, such as propane, which can be obtained in practically pure form free from contamination by naphthenes.

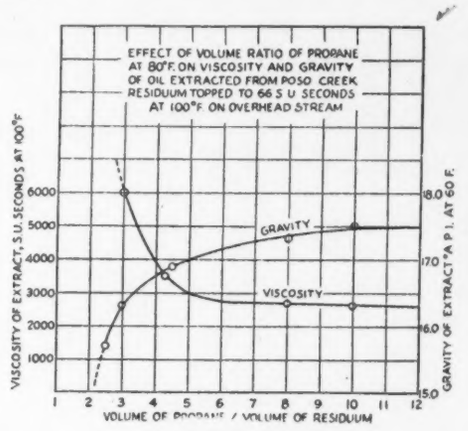

EFFECT OF VOLUME RATIO OF PROPANE AT 80°F. ON VISCOSITY AND GRAVITY OF OIL EXTRACTED FROM POSO CREEK RESIDUUM TOPPED TO 66 S U SECONDS AT 100°F. ON OVERHEAD STREAM
VISCOSITY OF EXTRACT, S.U. SECONDS AT 100°F.
GRAVITY OF EXTRACT A.P.I. AT 60 F.
GRAVITY
VISCOSITY
VOLUME OF PROPANE / VOLUME OF RESIDUUM

**FIGURE 3**

The report of Bahlke, Giles, and Adams[3] covers quite thoroughly a number of the most salient features of dewaxing with propane, particularly with respect to filtration. We may state that the results reported by the foregoing authors are in good agreement with our data. We shall, therefore, describe the dewaxing process only briefly—inserting some additional information that may be of interest.

In dewaxing with propane, refrigeration to about minus 40°F. is readily obtained by releasing the vapor at a controlled rate corresponding to lowering the temperature at the rate of 2 to 3°F. per minute. The low viscosity of the chilled mixture in combination with the favorable physical structure of the precipitated wax gives a readily filterable mixture with lubricating dis-

**TABLE 3**

***Dewaxing by Filtration from Propane Solution at —40 Deg. F.***

| SAMPLE | Gravity Deg. A.P.I. at 60° F. | Pour Point (Deg. F.) | Yield Percent by Volume of Stock | Viscosity, Saybolt Universal, at 210° F. | Volume of Propane Per Volume of Oil |
|---|---|---|---|---|---|
| Stock.....De-asphaltized Kettleman Hills long residuum | 19.8 | 90 | ... | 98 | ... |
| Dewaxed.....De-asphaltized Kettleman Hills long residuum | 18.5 | 5 | 83.2 | 105 | 3.0 |
| Stock.....De-asphaltized Santa Fe Springs long residuum | 20.1 | 90 | ... | 93 | ... |
| Dewaxed.....De-asphaltized Santa Fe Springs long residuum | 19.2 | 0 | 84.0 | 102 | 3.0 |
| Stock.....De-asphaltized Santa Fe Springs cylinder stock | 16.1 | 90 | ... | 390 | ... |
| Dewaxed.....De-asphaltized Santa Fe Springs cylinder stock | ... | 40*a* | 85.5 | 450 | 3.0 |
| Stock.....Santa Fe Springs distillate | 21.6 | 90 | ... | 84 | ... |
| Dewaxed.....Santa Fe Springs distillate | 20.6 | 10 | 87 | 86 | 3.0 |
| Stock.....Hydrolube from Santa Fe Springs distillate | 32.4 | 90 | ... | 59 | ... |
| Dewaxed.....Hydrolube from Santa Fe Springs distillate | 31.3 | 15 | 78 | 61 | 3.0*c* |
| Stock.....Mid Continent long residuum | 20.0 | 90 | ... | ... | ... |
| Dewaxed.....Mid Continent long residuum | 21.0 | 10 | 79.9*b* | 120 | 3.5 |
| Stock.....Mid Continent overhead cylinder stock raffinate | 28.3 | 90 | ... | 106 | ... |
| Dewaxed.....Mid Continent overhead cylinder stock raffinate | 26.7 | 5 | 73.6 | 117 | 3.0 |
| Stock.....Pennsylvania cylinder stock | 24.5 | 65 | ... | 185 | ... |
| Dewaxed.....Pennsylvania cylinder stock | 24.2 | 15 | 81*d* | 181 | 3.0*c* |

*a.* Viscosity pour point.
*b.* Yields of asphalt and wax were 5.4 and 14.7 percent respectively.
*c.* Wood-flour pre-coat, ¼-in.
*d.* Cake unwashed.

TABLE 4

*Acid Treatment of Propane De-asphaltized and Dewaxed Kettleman Hills Long Residuum with and without the Presence of Propane at 75 Deg. F.—15 Lb. 98 Per Cent Acid Per Barrel of Stock, Caustic-Soda and Water Washed, Blown Bright*

| SAMPLE | Volume Percent of propane | Yield Volume (Percent) | Gravity Deg. A.P.I. at 60 Deg. F. | Flash and Fire Points C.O.C. (Deg. F.) | Pour Point Deg. F.) | Carbon Residue (Per Cent) | Color N.P.A. | Acid No. | Viscosity, Saybolt Universal, at: 100 Deg. F. | Viscosity, Saybolt Universal, at: 210 Deg. F. | Viscosity-Gravity Constant | Viscosity Index |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Stock..... | .... | 100 | 18.3 | 450<br>525 | Fluid at 0 | 2.35 | Green<br>Opaque | 0.4 | 2,350 | 107 | 0.878 | 44 |
| Neutral oil.. | None | 72.1 | 19.8 | 450<br>525 | 0 | 2.17 | Opaque | 0.15 | 2,130 | 102 | 0.866 | 42 |
| Neutral oil.. | 300 | 82.0 | 20.8 | 445<br>510 | 5 | 1.28 | 7½ | 0.06 | 1,665 | 92.5 | 0.861 | 47 |

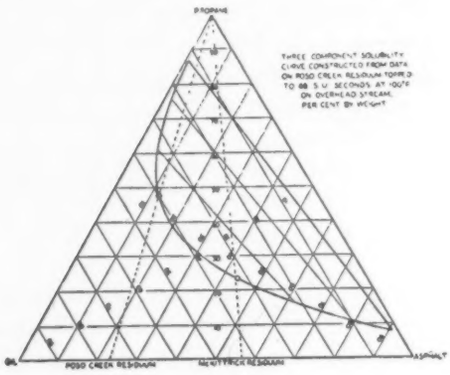


FIGURE 4

tillates, while the low specific gravity and low viscosity of liquid propane also facilitate the cold settling of the wax from heavy residual oils, and make possible a continuous cold-settling process. Filtration from propane solution is successfully carried out using ordinary No. 6 cotton canvas for the covering on the filter leaves, with or without the aid of a pre-coat of filter aid. Pour points as low as minus 25°F. have been obtained on distillate stocks, and pour points in the neighborhood of 0°F. can readily be obtained on a great many stocks.

Table 3 is presented to illustrate some of the stocks which have been dewaxed by filtration with propane. It should be pointed out that the volume ratio of propane can generally be reduced to as low as 1.5 volumes of propane for 1.0 volume of waxy oil containing 12 percent to 18 percent wax. The effect of changing the propane ratio on a particular stock is to increase the filtration rate of the propane solution; but the net filtration rate of dewaxed oil is, in general, not materially improved by increasing the propane ratio beyond about 2.0 volumes of propane to 1.0 volume of waxy stock.

As stated above, the dewaxing of heavy residual stocks is successfully accomplished by cold settling from propane solution. In this operation the minimum volume ratio of propane consistent with a sufficiently high settling rate to permit a continuous operation is rather critical, as is to be expected from consideration of specific-gravity and viscosity changes with increasing propane ratio—being in the neighborhood of 3.5 volume of propane to 1.0 volume of waxy oil for the majority of the untreated residual stocks. The simultaneous precipitation of small amounts of asphaltic and high-molecular-weight resinous materials along with the wax is not objectionable, and actually appears to serve a useful purpose in aiding the agglomeration and settling characteristics of the precipitated wax particles.

Distillate oils are more difficult to cold settle, requiring an appreciably higher propane ratio; but their settling is aided by the addition of high-molecular-weight or colloidal materials which tend to be precipitated in a viscous or plastic state at the low propane temperatures.

Figure 7 is presented to illustrate the effect of varying the propane ratio on the rate of settling at —40°F. of a waxy long residuum de-asphaltized with propane at 80°F.

For continuous cold settling of propane-de-asphaltized residual gas oils using a propane ratio of 4.0 to 1, a decantation rate of 12 inches per hour and a settling time of two hours appear to be adequate.

## ACID AND CLAY TREATING IN PROPANE SOLUTION

Acid treatment in propane solution, subsequent to asphalt and wax separation, is very effective for

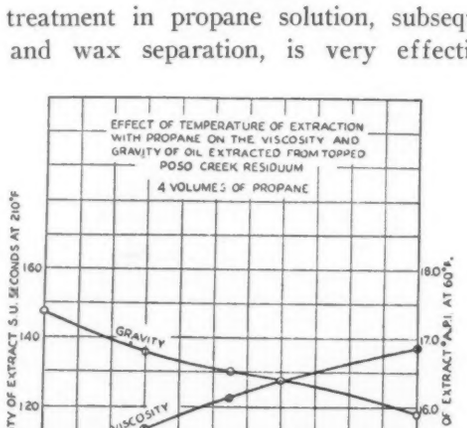



FIGURE 5

residual oils of the mixed-base type. The sludge separates cleanly, and is sufficiently mobile to be withdrawn from the bottom of the treating vessel—apparently due to a small amount of dissolved propane. Alkali wash-

ing, to remove organic acidity and any traces of suspended inorganic acidity, is easily accomplished in propane solution with practically no tendency toward emulsification with even high-speed agitation. An advantage in acid treating in propane solution is that the yield is increased and the consumption of acid reduced for the same color and carbon residue on the treated oil.

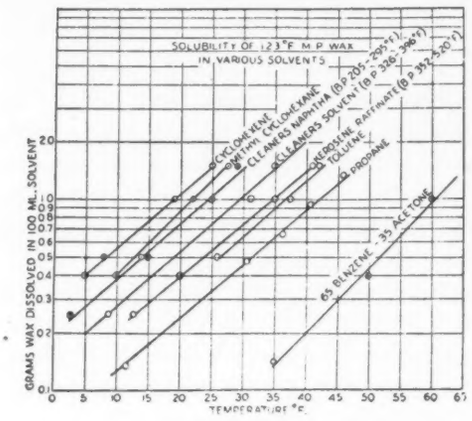



FIGURE 6

To illustrate the efficiency of acid treating in the presence of propane, Table 4 shows the results of comparable acid treatments on propane-de-asphaltized and dewaxed (continuous cold settling) Kettleman Hills long residuum followed by alkali and water washings, with and without the presence of propane. The marked improvement in yield and color is apparent from the table, and the appearance of the finished oil is decidedly in favor of the oil treated in the presence of propane, which shows an excellent green fluorescence without clay treatment.

Clay percolation, while the oil is still dissolved in the propane solution, has been found to be very efficient at atmospheric temperatures and higher.

## COMBINATION OF PROPANE WITH SELECTIVE SOLVENTS

One of the outstanding uses for propane, which was found in the early stages of the investigation, is as a diluent for the lubricating stock during the use of selective solvents for removing the aromatic, unsaturated, and naphthenic fractions. In considering such use of propane, we shall refer to the other solvent which has a greater miscibility with aromatic and naphthenic than with paraffinic hydrocarbons as the selective solvent; because lubricating stocks containing aromatic, naphthenic, and paraffinic fractions are readily miscible with propane, except for the asphaltic bodies discussed in the first part of this paper. Experience has shown that propane alone as a solvent cannot be used to make a separation between aromatic, unsaturated, and naphthenic lubricating fractions on the one hand, as compared with the paraffinic lubricating fractions on the other; but when propane is used in conjunction with a selective solvent which is practically insoluble in propane, a better control can generally be obtained over the behavior of the selective solvent. Among other things, the action of propane appears to be to suppress

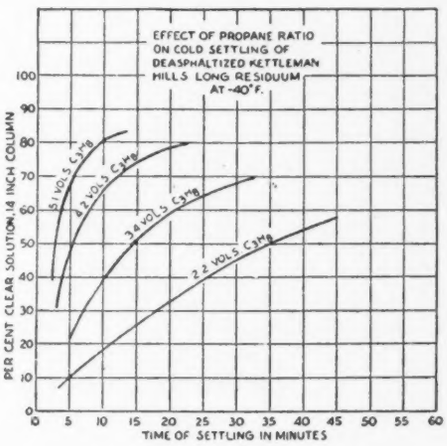

EFFECT OF PROPANE RATIO ON COLD SETTLING OF DEASPHALTIZED KETTLEMAN HILLS LONG RESIDUUM AT -40°F.
1.1 VOLS. C3H8
2.2 VOLS. C3H8
3.4 VOLS. C3H8
2.2 VOLS. C3H8
PER CENT CLEAR SOLUTION, 14 INCH COLUMN
TIME OF SETTLING IN MINUTES

FIGURE 7

TABLE 5

*Treatment of Kettleman Hills Dewaxed and De-asphaltized Long Residuum with Nitrobenzene with and without the Presence of Propane*

| SAMPLE | Treatment: Nitrobenzene (Per Cent by Volume) | Treatment: Propane (Per Cent by Volume) | Treatment: Temperature (Deg. F.) | Tests on Raffinate After 5 Percent Death Valley Clay at 325 Deg. F.: Yield Volume (Per Cent) | Gravity Deg. A.P.I. | N.P.A. | Carbon Residue (Per Cent) | Viscosity Saybolt Universal at: 100 Deg. F. | Viscosity Saybolt Universal at: 210 Deg. F. | Viscosity-Gravity Constant | Viscosity Index |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Stock | ..... | ... | .. | 100 | 18.6 | opaque | 2.08 | 2,030 | 97 | 0.876 | 33 |
| Raffinate No. 1 | 3×100 | None | 37 | 26.10 | 27.4 | 8½+ | 0.36 | 774 | 75.5 | 0.815 | 92 |
| Raffinate No. 2 | 3×100 | None | 37 | 23.30 | 27.4 | 8½+ | 0.38 | 770 | 75.5 | 0.815 | 92 |
| Raffinate No. 3 | 3×100 | None | 37 | 10.36 | 28.4 | 7 | 0.21 | 820 | 82 | 0.806 | 101 |
| | 2×100 | None | 68 | | | | | | | | |
| Raffinate No. 4 | 3×100 | None | 37 | 4.73 | 29.4 | 6½ | 0.21 | 1,060 | 95.5 | 0.796 | 105 |
| | 3×100 | None | 68 | | | | | | | | |
| Raffinate No. 1P | 3×100 | 300 | 37 | 55.10 | 26.0 | ... | 0.29 | 670 | 69.5 | 0.827 | 85 |
| Raffinate No. 2P | 3×100 | 300 | 37 | 51.80 | 26.0 | 6½ | 0.21 | 662 | 68.5 | 0.828 | 83 |
| Raffinate No. 3P | 3×100 | 300 | 37 | 29.40 | 29.3 | 5 | 0.11 | 450 | 62 | 0.806 | 102 |
| | 3×100 | *a* | 68 | | | | | | | | |
| Raffinate No. 4P | 3×100 | 300 | 37 | 19.2 | 29.8 | 5 | 0.08 | 466 | 63.5 | 0.803 | 106 |
| | 3×100 | *a* | 72 | | | | | | | | |

a. Experiment continued without removal or further addition of propane.

the solubility of the selective solvent in the raffinate phase, making for greater selectivity of the solvent, and at the same time to facilitate a better segregation of the colored and carbon-forming bodies into the extract phase. In other words, a very marked advantage of employing selective solvents in the presence of propane is an increased yield of raffinate of a given quality along with lower N.P.A. colors and carbon residues on the finished raffinate. The amount of the selective solvent required to give a raffinate of a definite quality may be slightly greater than when propane is not used, but the benefits of increased yield and saving on subsequent treatment are often more than sufficient to justify the use of propane with a suitable solvent.

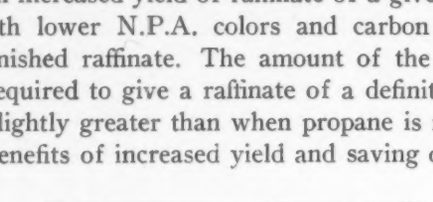



**FIGURE 8**

As an example of the use of propane in combination with selective solvents to remove aromatic, unsaturated, and naphthenic fractions, Table 5 and Figure 8 have been constructed from data on nitrobenzene treatment of propane-de-asphaltized and propane-dewaxed Kettleman Hills long residuum. The first group of experiments shown in the table was performed without propane being present, using external refrigeration; whereas the second group was performed with the stock dissolved in propane and with internal refrigeration. All experiments shown were performed with a multiple-batch method, in which the stock is re-treated with a fresh portion of solvent.

In comparing the results of such extraction experiments, comparisons should be made between raffinates of the same viscosity index or viscosity-gravity constant. Thus, it is seen that for a viscosity index of 92 or a viscosity-gravity constant of 0.815, the yield of raffinate without propane was 24 percent; whereas with propane the yield was 45 percent of raffinate, with only about half the carbon residue and true color concentration. When the treatment is carried further, to give a viscosity index of 102 or a viscosity-gravity constant of 0.806 in both cases, the improvement in yield is even more pronounced—the yields being 11 percent without propane and 29 percent with propane—and the color and carbon residues are still distinctly better when propane is used. It should be pointed out that with continuous counter-current treatment the yields of raffinate of a given quality can be expected to be appreciably greater than in the multiple-batch experiments shown above, and at the same time the amounts of selective solvent required will be materially reduced.

It will be noted that the viscosities of the raffinates are lower when propane is used; but there is evidence to show that this is not due mainly to the removal of an excessive amount of high-molecular-weight fractions when propane is present, but rather to the removal of an excessive amount of low-molecular-weight fractions when propane is absent.

A better understanding of the function of propane when used in conjunction with selective solvents is obtained from a consideration of the phase relationships in the extraction with a selective solvent which is practically immiscible with propane. With certain selective solvents such as cresol, we find that it is impossible to obtain a satisfactory phase separation at any reasonable temperature without the presence of propane when the stock shows a low degree of paraffinicity, as in the case of de-asphaltized, dewaxed Kettleman Hills long residuum of 0.878 viscosity-gravity constant. In other words, this particular stock and selective solvent are miscible in all proportions at the temperatures available from a practical standpoint; but it should be constantly borne in mind that whether a stock of a given average molecular weight will show complete miscibility with the selective solvent at the available temperatures is determined by the paraffinicity of the stock. If propane is added to a one-phase system consisting of such a selective solvent and stock in suitable proportions we find by experiment that a point will be reached at which a separation of a second phase occurs, and that the amount of propane required to cause phase separation is dependent on the paraffinicity of the original stock. It is also found that the same quantity of propane is required in a batch extraction to give a satisfactory separation of phases, regardless of whether the stock is dissolved in propane before contacting with the solvent or whether the propane is added after mixing the oil and solvent—provided, of course, that the agitation is sufficient to insure equilibrium between phases in all cases. On account of the ready miscibility of propane with the lubricating stock and its immiscibility with the selective solvent, the propane solution of lubricating stock actually constitutes the stock being subjected to extraction by the selective solvent.

In the foregoing example, in which no phase separation can be obtained with the selective solvent on the original stock, it may be considered that one function of propane added is to increase the paraffinicity of the combined stock until a satisfactory separation of phases is obtained; but a peculiar advantage of propane as compared to other higher-molecular-weight paraffinic

diluents which might be added, or re-cycled, appears to reside in its extremely low solvent power for certain selective solvents. This particular feature of propane reduces markedly the concentration of the selective solvent in the stock or raffinate phase, and thereby increases the effective selectivity of the solvent as regards segregation into the extract phase of only the materials which are most readily soluble in the selective solvent, viz., the most aromatic and naphthenic fractions responsible for dark colors and high carbon residues. For example, the concentration of nitrobenzene was determined in the raffinate phases as they were taken from the extraction apparatus in the experiments shown in Table 5. Without propane the concentration of nitrobenzene in the raffinate phase was about 18 percent to 20 percent by volume; whereas in the presence of propane the corresponding value was approximately eight percent.

From the standpoint of practical operation, the low viscosity and low specific gravity of the raffinate phase (propone phase) aids materially in obtaining adequate contact and equilibrium between phases, and in the coalescence of the respective phases before separating. Also in the case of solvents requiring a low temperature for satisfactory operation, as for example a mixture of cresols, refrigeration by means of propane is readily accomplished by admixing the stock with propane and refrigerating by evaporation of a portion of the propane—with separation of any precipitated asphaltic phase before introducing the stock into the counter-current treating system. The solvent is precooled in a similar manner if desired.

The solvent treatment of waxy stocks in propane solution may be accomplished at temperatures either above or below that at which wax begins to precipitate in the propane solution, but it is possible with a number of solvents to operate at a sufficiently high temperature to keep all of the wax in solution during the solvent treatment.

The proportion of propane employed in solvent extraction is often insufficient, with residual oils of low asphalt content, such as the better grades of Mid-Continent oils, to cause precipitation of a sufficient quantity of an asphalt phase to justify removal by settling prior to solvent treatment; and the propane solution of such oils may be introduced directly into the solvent-treating system. With some of the more selective and effective solvents, the asphalt is completely removed into the extract phase, and a raffinate of satisfactory color is obtained without subsequent acid treatment. The finishing treatment in such cases would usually consist of a hot contact-clay treatment employing five percent to 10 percent of decolorizing clay.

The authors wish to express their appreciation of the helpful suggestions and enthusiastic support received from R. E. Haylett and D. R. Merrill in the course of the research reported in this paper, and to acknowledge the assistance of the many laboratory co-workers, particularly R. C. Pollock, M. L. Wade, and V. N. Jenkins.

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# Prospects of a Petroleum Chemical Industry

(*Continued from page* 339)

by esterification with rosin or by interaction with phenol alcohols, and in the manufacture of lacquers and plastics.

### ETHYLENE DICHLORIDE

Ethylene dichloride is made by the direct addition of chlorine to ethylene or to the ethylene fraction of cracking gas. With dry gases and at ordinary temperatures, reaction is rather slow. It is much faster when carried out in the liquid phase, in the presence of catalysts or of solvents, or at high temperatures. Under the last named conditions it is difficult to limit the reaction, since substitution products are also formed. Ethylene dichloride has been suggested as a solvent for this reaction. Another method is to mix liquid chlorine with ethylene at 0° C. (32° F.). The dihalide is also prepared by allowing a mixture of chlorine and ethylene to flow through a reaction tower countercurrent to a stream of water.

Ethylene dischloride is a colorless liquid ($d_4^{20}$ 1.2569), boiling at 83.5° C. (182.3° F.) and freezing at —36° C. (— 32.8° F.), slightly soluble in water, more so in alcohol and ether, and miscible with oils and fats. It burns with difficulty and the flames are readily extinguished. Because of resistance to hydrolysis and oxidation, this substance may be used at its boiling point with little danger of corrosion. Slow hydrolysis on exposure to light is retarded by addition of a small proportion of alcohol.

This diahalide is employed as a fumigant, as a solvent, and in the manufacture of ethylene glycol. As a solvent ethylene dichlordide can be used in the extraction of oils, fats, and resins, and, mixed with methyl or ethyl alcohol, will dissolve cellulose nitrate or acetate. Another application is in the dewaxing of lubricating oil fractions, since paraffin wax is only slightly soluble in this solvent at temperatures below 25° C. (77° F.). Either alone or mixed with carbon tetrachloride it is a good cleansing agent. On a volumetric basis ethylene dichloride is a cheaper solvent than carbon tetrachloride and superior in stability towards water and steam.

An interesting product derived from ethylene chloride is that designated by the term "Thiokol." The latter is made by the interaction of the dihalide with polysulfides of alkali or alkaline earth metals. Thiokol resembles soft rubber in properties but is characterized by a high sulfur content and insolubility in practically all organic liquids. It can be incorporated with crude rubber and the mixture vulcanized. The resulting products are also insoluble in organic liquids, particularly petroleum distillates, and are used, for example, in materials such as hose for handling gasoline.

### GLYCOLS

Hydrolysis of ethylene dichloride or of ethylene chlorohydrin with solutions of alkalies leads to the formation of ethylene glycol. Direct oxidation of ethylene to glycol in the presence of water has also been employed. For example, ethylene and oxygen are introduced into water containing iodine (or compounds which readily liberate iodine, such as potassium triiodide or ferric iodide) and a manganese or iron oxidation catalyst. Or ethylene is allowed to react with air and water at 150° to 300° C. (302° to 572° F.) under pressure in the presence of metallic catalysts.

Ethylene glycol is a colorless, odorless liquid which possesses a sweet taste, boils at 197.5° C. ( 387.5° F.), and has a density of 1.115 at 20° C. (68.° F.). It is miscible in all proportions with water, alcohol, and many other solvents.

Possibly the most common uses of glycol are as a solvent and an antifreeze for automobile radiators. In addition, it is employed in the manufacture of cosmetics, in the preservation of anatomical and biological specimens, in the treatment of skins and furs, and as an aid in the finishing and dyeing of fabrics. Derivatives of ethylene glycol, however, have found diverse industrial applications. Thus, the monoethers (e. g., the monoethyl ether or Cellosolve) are widely used in lacquers. The nitrates form the basis for low-freezing dynamites. Esters of glycols and polybasic acids (e. g., phthalic) are of resinous nature and are useful in lacquers.

Higher boiling materials, the so-called polyglycols, are formed as by-products during the making of glycols. These substances can be made also by dehydration of ethylene glycol or by the action of water on ethylene oxide. The lowest polyglycol, diethylene glycol, has proved to be an almost ideal lubricant for wool spinning. Various derivatives of polyglycols, such as the monoethyl ether of diethylene glycol or Carbitol, have found varied applications.

### OLEFIN OXIDES

Compounds of the olefin oxide type, although classed as neutral substances, are capable of taking part in an astonishing number of reactions, and interest is centered chiefly in their derivatives rather than in the oxides themselves. Thus far only ethylene oxide appears to be utilized commercially, although higher olefin oxides are known. These oxides are made by heating either chlorohydrins with solid

or anhydrous alkalies or concentrated solutions of chlorohydrins with alkalies. Ethylene oxide is said to be obtained on conducting ethylene, steam, air, and hydrogen over metallic catalysts at 150° to 400° C. (302° to 752° F.).

Ethylene oxide (and alkalene oxides generally) reacts with organic substances containing an easily replaceable hydrogen atom—e. g., alcohols, acids, phenols, and amines—and also with water. These reactions may be represented as:

$$\begin{matrix} CH_2 \\ | \\ CH_2 \end{matrix}\!\!>\!O + HR \rightarrow \begin{matrix} CH_2OH \\ | \\ CH_2R \end{matrix}$$

Glycol monoethers are made by the reaction of ethylene oxide, or other aliphatic oxide, with an alcohol:

$$\begin{matrix} CH_2 \\ | \\ CH_2 \end{matrix}\!\!>\!O + C_2H_5OH \rightarrow \begin{matrix} CH_2OC_2H_5 \\ | \\ CH_2OH \end{matrix}$$

Catalysts for the above reaction include sulfuric acid, the sulfates of nickel, zinc, or chromium, hydrosilicates of aluminum, and dialkyl sulfates. When an excess of the olefin oxide is employed, it is possible to make polyolefin glycol ethers. For example, introduction of ethylene oxide into anhydrous ethyl alcohol containing a small amount of sodium (as a catalyst) leads to the formation of the ethyl ether of tetraethylene glycol and higher polyglycols. The monoalkyl ethers of ethylene glycol are solvents for cellulose nitrate, resins, gums, and oils. They are miscible with organic solvents, and the lower ethers are soluble in water. The methyl ether is a solvent for cellulose acetate, and is especially adapted to the preparation of lacquers.

## ETHANOLAMINES

Ethylene oxide reacts with aqueous ammonia to yield a mixture of three basic substances, mono-, di-, and triethanolamines, which can be separated by distillation. The proportions of the three products depend largely on the relative proportions of the reactants, an excess of ethylene oxide favoring the formation of triethanolamine. The boiling points of the ethanolamines are are follows:

| Ethanolamines | Temp. ° C. (° F.) | Pressure Mm. (in.) |
|---|---|---|
| Mono-, $NH_2CH_2CH_2OH$ | 171 (339.8) | 757 (29.8) |
| Di-, $NH(CH_2CH_2OH)_2$ | 217 (422.6) | 150 ( 5.9) |
| Tri-, $N(CH_2CH_2OH)_3$ | 277 (530.6) | 150 ( 5.9) |

These substances are miscible in all proportions with water, alcohol, acetone, glycerol, and ethylene chlorohydrin, but not with ether and some aldehydes.

Monoethanolamine is a colorless, slightly viscous, hygroscopic liquid, with a faint ammoniacal odor. Diethanolamine is a colorless, odorless liquid, with viscosity characteristics similar to those of glycerol. The dinitrate of monoethanolamine and the trinitrate of diethanolamine have been proposed as explosives. Pure triethanolamine is a white crystalline solid at room temperature. The commercial product is a clear, viscous, hygroscopic liquid with a density of 1.124 at 20° C. (68° F.) soluble in most organic liquids containing oxygen, but only slightly soluble in hydrocarbons.

In the Girbotol process of the Girdler Corporation aqueous solutions of a mixture of the ethanolamines, particularly the di- and triethanolamines, are employed for the removal of carbon dioxide and hydrogen sulfide from gases. The resulting carbonates and acid sulfides readily decompose at temperatures above 50° C. (122° F.) with regeneration of the solvent. The ethanolamines form stable salts with sulfur dioxide and therefore are unsatisfactory for the removal of that gas.

Other uses have been suggested for these amines —e. g., as extraction agents in the refining of mineral oils, plasticizers for cellulose esters, catalysts in the formation of phenol-aldehyde condensation products, preservative agents in rubber compositions, and stabilizers for tetraalkyl leads.

Ethanolamines combine readily with fatty acids to yield soaps, whose consistency depends primarly upon the acid employed. The oleate resembles petroleum jelly, whereas the stearate is a hard, waxlike solid. These compounds are soluble in water, benzene, toluene, and turpentine, but only slightly so in lubricating and petroleum oils. In contrast with the usual sodium or potassium soaps, those of the ethanolamine type are characterized by low alkalinity. The oleate may be used for promoting oil-in-water emulsions.

## DIOXAN

Diethylene dioxide, or dioxan $O{-}CH_2CH_2{-}O{-}CH_2CH_2$, although structurally related to ethylene oxide, should more properly be considered as a derivative of ethylene glycol. It is made industrially by heating ethylene glycol with dehydrating agents, such as sulfuric acid or anhydrous ferric sulfate. Similarly, dimethyldioxan is obtained from 1,2-propylene glycol, and diethyldioxan from 1,2-butylene glycol.

Dioxan is a liquid boiling at 102° C. (215.6° F.) and melting at 11° C. (51.8° F.). It is soluble in water and in many of the usual organic solvents, and is a solvent for resins, fats, and cellulose esters.

## PRODUCTION OF UNSATURATED HYDROCARBONS

The action of heat on saturated hydrocarbons results in the formation of unsaturated compounds and also in many instances leads to a rearrangement within the molecule. Perhaps the largest source of unsaturated hydrocarbons is cracking gas, the composition of which has already been discussed. Butadiene is a component of vapor-phase cracking gas. Acetylene is formed from hydrocarbons at temperatures considerably above those employed in cracking. These two unsaturated compounds are of particular interest.

## ACETYLENE

Acetylene is one of the thermal decomposition products of hydrocarbons at 800° C. (1472° F.) or above. Below this temperature it is not formed, or only in small proportions (methane, for example, requires heating to 1000-1200° C. or 1832-2192° F.). Acetylene is made by the thermal decomposition of practically all hydrocarbons in the electric arc. Generally, better yields are obtained from low-boiling or gaseous petroleum hydrocarbons than from higher boiling distillates. Short periods of heating, reduced pressure, and addition of hydrogen or steam to the gaseous hydrocarbons minimize their decomposition to carbon and hydrogen.

One important reaction of acetylene is its hydration to acetaldehyde, which may be subsequently oxidized to acetic acid. Hydration can be conducted in either the liquid or gaseous phase. In liquid-phase operations, a stream of acetylene is passed through dilute sulfuric acid (about 6 percent) to which has been added mercuric oxide, the mercury salt thus formed serving as a catalyst. Reaction proceeds with the evolution of heat, and vaporized acetaldehyde is carried along in the gas stream from which it is recovered by cooling. Oxidation of the aldehyde readily yields acetic acid. A number of by-products are formed in minute proportions in the hydration of acetylene. One of these, diacetyl, is used for flavoring butter substitutes.

A difficulty encountered in vapor-phase operations is the ease with which acetaldehyde is converted to other substances, such as crotonaldehyde or esters. This is overcome by the addition of a gaseous oxidizing agent—e. g., by conducting acetylene, steam, and oxygen at 100° to 200° C. (212° to 392° F.) over a catalyst consisting of a mercuric salt.

Acetylene is being produced at a refinery in this country by arcing hydrocarbon gases and is used subsequently for making acetic acid. Thus petroleum has definitely become a source of another hydrocarbon, acetylene.

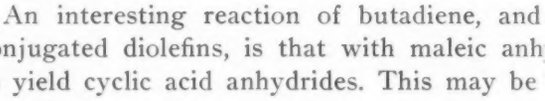
Other important derivatives of acetylene are vinyl esters and acetylene polymers. Vinyl esters are obtained from acetylene and acids by direct addition in the presence of a mercury catalyst. Vinyl esters can be transformed by various methods into light-colored resins (Vinylite) which at present are finding rather wide utilization in coatings and plastics, particularly in phonograph records.

Of the various polymers of acetylene, one is the result of the action of heat on the hydrocarbon in contact with certain metals. This is known as cuprene and is an insoluble, brown, amorphous solid. Liquid polymers are made by absorption of acetylene in solutions of cuprous salts. Mono- and divinylacetylenes are thus formed. Divinylacetylene slowly thickens to a viscous liquid which, after application as a film, hardens farther to an insoluble coating. Monovinylacetylenes, when combined with hydrochloric acid, yields the liquid called "chloroprene." The latter can be changed readily into an elastic solid which resembles natural rubber more closely than does any other synthetic material. Acetylene rubber of this type is known as DuPrene.

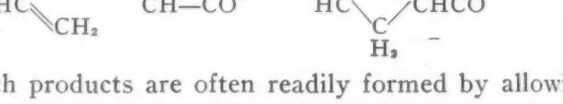
## BUTADIENE

Butadiene is a gaseous product of the pyrolysis of hydrocarbons at temperatures just below those at which transformation of straight-chain hydrocarbons to aromatics takes place. To illustrate, the maximum yield of butadiene from ethylene is obtained at 750° C. (1382° F.), and from propane, propylene, and butane at about 727° to 728° C. (1340.6° to 1342.4° F.). Cyclohexane is converted largely to butadiene at 654° C. (1209.2°F.).

An interesting reaction of butadiene, and other conjugated diolefins, is that with maleic anhydride to yield cyclic acid anhydrides. This may be represented as:



Such products are often readily formed by allowing the diolefin and maleic anhydride to react in the presence of benzene. Not only aliphatic but also cyclic diolefins—e. g., cyclopentadiene—combine with maleic anhydride:



By this reaction it was shown that butadiene, piperylene, isoprene, and cyclopentadiene were present in the low-boiling fractions of a gasoline cracked at a high temperature.

Butadiene and similar diolefins have been considered for some time as a source of artificial rubber. When heated with various substances which act as catalysts, butadiene polymerizes and forms a solid mass possessing some of the properties of rubber. Considerable work has been done recently on this reaction, but the development is hampered by economic considerations. However, a resin has been produced from the diolefins of vapor-phase-cracked distillates by polymerization with aluminum chloride. This petroleum resin produces varnishes which dry very rapidly. Another related material is a dark colored liquid product consisting of a mixture of polymerized olefins and diolefins, known as Puresinol. This is a by-product in the refining of highly cracked gasoline distillates and is proposed as a substitute for linseed and other drying oils. Its specific gravity is 0.956, Saybolt viscosity at 100° F. (37.8°

C.) is 200 seconds, and iodine number is 175. Brittleness of the dried film would appear to limit its uses.

## AROMATICS

Certain crudes, particularly Borneo petroleum, contain aromatic hydrocarbons to some extent. Aromatics are also formed in cracking processes. However, in the production of Hydrosolvents by hydrogenation it has been found that aromatic compounds are produced in large proportions.

If an overhead cut is made of a Hydrosolvent, the following results roughly express the proportion of aromatic hydrocarbons:

| ° F. | (° C.) | % |
|---|---|---|
| 200-275 | (93.3-135) | 10 |
| 275-365 | (135-185) | 35 |
| 365-419 | (185-215) | 38 |
| 419-460 | (215-237.8) | 8-10 |

Distillate under 200° F. (93.3° C.) end point is added to gasoline.

The increase in aromatic content with boiling point is of significance in the coating industry, since a coating in which such a liquid is present as the thinning agent becomes richer in more powerful solvents as evaporation increases. This tends to permit the binder in the composition to orient satisfactorily, giving an unusually hard and glossy surface.

In the crude Hydrosolvent cut there is probably about 1 percent benzene, 4 percent toluene, 18 percent xylenes, and 25 percent of the trimethylbenzene type of hyrocarbons, including mesitylene and isomers, such as methylethylbenzene propylbenzine etc. There are also indications of the presence of hydrogenated naphthalenes, particularly tetralin and decalin. There may be also some octalin. Although some naphthalene may be in the distillates, it is likely to be negligible in amount.

The hydrocarbons from coal tar are characterized by the insignificant proportions of trimethylbenzene hydrocarbons, and the high yield of these through hydrogenation of petroleum appears to fill a gap not supplied by coal tar. In other words, the hydrocarbons of coal tar are benzene, toluene, and xylene, and then rather suddenly there is a jump to naphthalene, anthracene, and other polycyclic hydrocarbons. Hydrogenation, therefore, should serve as a means to place the petroleum industry in the coal-tar- field, for products in which coal tar is lacking.

## OXIDATION PRODUCTS

The oxidation of petroleum hydrocarbons of high molecular weight, such as paraffin wax, is a method for the preparation of acids of high molecular weight. Air, oxygen, ozone, oxides of nitrogen, and nitric acid can be used as the oxidizing agents, though air is most generally employed. Reaction is carried out at temperatures above the melting point of the wax, usually about 160° to 180° C. (320° to 356° F.). Catalysts include metallic oxides, inorganic salts of organic acids (e. g., barium stearate or calcium cinnamate), manganese acetylacetonate, and naphthenate.

Separation of the acids from the reaction product is accomplished by extraction with alcohols or with alkalies, by sweating—i. e., partial liquefaction of the product by heat followed by pressing to separate the liquid and solid portions—and by distillation under reduced pressure.

These acids are designated "wax acids," as this term indicates wax as their source rather than fats. Furthermore, some investigators have intimated that these acids contain a large proportion of substances having an uneven number of carbon atoms per molecule as contrasted with fatty acids which contain an even number of carbon atoms. Wax acids can be employed in the manufacture of synthetic fats by esterification with glycol or glycerol and for making soaps. The latter is the more important use. Crude wax acids are characterized by the presence of hydroxyl and aldehydic groups. the removal of these groups, which interfere in certain applications, is a problem to which considerable attention is being given. Instead of wax acids, higher alcohols can be obtained by employing the proper catalyst. Sulfation of these wax alcohols forms soaps of the hard-water type, a procedure which appears cheaper than making detergents from the alcohols of vegetable-oil hydrogenation.

In addition to acids of high molecular weight, water-soluble acids of low molecular weight are also obtained by oxidation, as well as intermediates such as aldehydes, ketones, esters, acid anhydrides, and hydroxy acids. In preparing alcohols, the wax is oxidized for a shorter time than that employed for making acids. Catalysts which are particularly useful in this reaction are acetic acid or weak inorganic acids such as boric. The alcohols are separated from nonoxidized wax and other substances by distillation under reduced pressure with steam.

Oxidized kerosene fractions are used for the production of an alcohol denaturant known as Aldehol.

## CHLORINATION PRODUCTS

Although a vast amount of work has been conducted on the chlorination of petroleum hydrocarbons, only brief mention can be made of some of the products obtained.

Chlorination of methane, for example, yields methyl chloride ($CH_3Cl$), methylene chloride ($CH_2Cl_2$), chloroform ($CHCl_3$), and carbon tetrachloride ($CCl_4$). Methyl chloride, because of its low boiling point, has been used as a refrigerant. The remaining three substances can serve as solvents, chloroform to a limited extent as an anesthetic, and carbon tetrachloride as a fire extinguisher. Carbon tetrachloride and antimony trifluoride react to give dichlorodifluoromethane, a substance particularly ap-

plicable as a refrigerant because of its low toxicity.

Chlorination of natural-gas gasoline gives a mixture of amyl chlorides which can be hydrolized to amyl alcohols (marketed under the name of Pentasol) and the latter esterified with acetic acid (Pentacetate.)

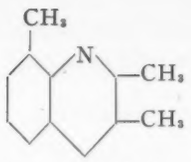
The production of Paraflow is an outstanding development in the utilization of chlorinated petroleum hydrocarbons. Paraffin wax is chlorinated until its chlorine content is about 10 to 12 percent and is then reacted with benzene, naphthalene, or anthracene in an inert solvent (e. g., kerosene) at 60° to 71° C. (140° to 160° F.), with aluminum chloride as a catalyst. After reaction is completed, the aluminum chloride sludge is withdrawn, and the solvent and excess wax are removed by distillation, the latter under reduced pressure. The product is a chlorine-free, synthetic hydrocarbon oil possessing the unusual property of lowering the pour point of wax-bearing lubricating oils. For example, the addition of 1 percent of this synthetic oil to a crude Pennsylvania lubricating oil reduced its pour point from —1.1° C. (+30° F.) to below —20.6° C. (—5° F.). The utilization of Paraflow, therefore, makes it possible to eliminate the severe dewaxing operations required in the refining of many lubricating oils.

## NITROGEN COMPOUNDS FROM PETROLEUM

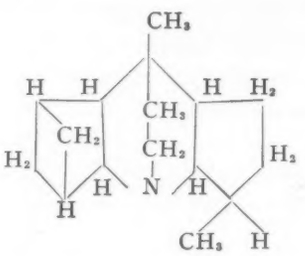
In coal oil and shale oil there are present aromatic compounds of nitrogen whose identification has been accomplished with comparative ease. Petroleum, however, contains an astonishing variety of nitrogen compounds of unknown structure, some of which, according to present indications, fall into the class of hydroaromatics and until recently were very little known. Hydroaromatics are closely related to alkaloids, and to change hydroaromatic bases to alkaloids it is necessary only to make the proper placement of oxygen. A brief mention is therefore made of these nitrogen compounds as opening up a new field of scientific interest and practical value, some of them already having been found useful in insecticides and as pickling inhibitors.

The nitrogen content of crude petroleum is rather low, about 0.1 percent or less, though some California oils have shown as high as 0.82 percent on analysis. Apparently the nitrogen compounds in crude oils are nonbasic and are not extractable with acids. Distillates from such crudes, however, contain nitrogen bases, thus indicating a decomposition of rather complex nitrogenous substances during distillation.

To illustrate the types of compounds which have been isolated, only two need be mentioned. Both of these were found in the sulfur dioxide extract obtained in the refining of kerosene. From the fraction boiling at 276° to 277° C. (528.8° to 530.6° F.) 2,3,8-trimethylquinoline was obtained:



Ahydroaromatic base, $C_{16}H_{25}N$, from the same fraction is of interest in that it represents a compound of the cyclopentane type. The structure assigned to this substance is:



This has been named decahydro-3,8-dimethyl-5,7-methano-4,8-ethanopyrindacine.

So far there is no explanation for the presence of hydroaromatics in petroleum and their absence from coal, shale, and bone distillates. Furthermore, none of the bases isolated from petroleum has been obtained from other natural sources.

## SULFUR COMPOUNDS FROM PETROLEUM

The sulfur content of crude oils from different localities varies widely, ranging from less than 0.2 percent to as high as 4 to 5 percent. Fractions of high-sulfur content are the basis of Alcotate, an alcohol denaturant. Practically all of the simpler types of sulfur compounds have been identified in such oils; these include mercaptans, thioethers, thiophenes, thiophanes, organic sulfides and disulfides, compounds containing both sulfur and oxygen, and also hydrogen sulfide, elemental sulfur, and carbon disulfide. From the refiner's viewpoint these sulfur-containing substances are impurities which, because of their corrosive properties or their odors, must be removed particularly from gasoline and kerosene fractions. To the chemist, the isolation and utilization of these sulfur compounds opens a field of wide possibilities.

## HYDROGEN SULFIDE

Hydrogen sulfide may be extracted from petroleum or its distillates, or from the gases from the distillation or cracking of crudes of distillates of high sulfur content, by solutions of alkalies—e. g., sodium hydroxide, sodium carbonate and bicarbonate, or ethanolamines. Aqueous suspensions of brucite have been suggested for the same purpose. Hydrogen sulfide may be liberated, except in the case of sodium hydroxide, by heating the solutions or suspensions.

The uses of gaseous hydrogen sulfide, other than in the laboratory, appear to be rather limited. It reacts at high temperatures with olefins or acetylene in the presence of catalysts to yield mercaptans,

thioethers, thiophene and its derivatives, and carbon disulfide. When hydrogen sulfide is mixed with methane, ethane, or propane, and the mixture is heated to 1200° C., both benzene and carbon disulfide are obtained. The salts of hydrogen sulfide, particularly the sodium salt, have been used as aids in dyeing, as reducing agents, depilatories, and denitrating agents, and in the manufacture of sulfur dyes and lithopone.

The preferential oxidation of hydrogen sulfide to sulfur, either by metallic oxides or by sulfur dioxide, are well-known processes and suggest the possibility of high-sulfur petroleum oils as potential sources of sulfur.

## MERCAPTANS

Aqueous solutions of sodium hydroxide and alcoholic solutions of sodium plumbite or of sodium or potassium hydroxide have been employed for the extraction of mercaptans from petroleum distillates. As an industrial method the use of aqueous sodium hydroxide is probably to be preferred, and mercaptans may be thus liberated by steam distillation.

Mercaptans have been suggested as pickling inhibitors and as intermediates in the preparation of dyes. Heated with phosphorus tri- or pentasulfide, mercaptans yield liquids which may be employed in ore-flotation processes. The well-known soporific, sulfonal, is the sulfone of the mercaptole obtained from acetone and ethyl mercaptan. Because of their penetrating odor, volatility, and insolubility in water, the lower boiling mercaptans, especially ethyl mercaptan, have been used for detecting leaks in gas mains. A product designated Cal-Odorant, apparently a mixture of hydrocarbons and sulfur compounds, is marketed for odorizing natural gas. Butyl mercaptan is said to be a stabilizer for chlorinated hydrocarbons—e. g., carbon tetrachloride or tetrachloroethylene.

## THIOETHERS

Possible methods for the recovery of thioethers include the formation of addition compounds with salts —e. g., mercuric iodide or chlorideg titanium tetrachloride, or platinic chloride—and their extraction from acid sludge. Passage of mercaptans over metallic catalysts (sulfides of cadmium or zinc, and aluminum chloride) at 320° to 350° C. (608° to 662° F.) results in the formation of thiothers.

Thioethers are solvents for rubber and for resins such as abietic acid, paracoumarone, petroleum resin, and cresol-sulfur chloride resins. Ethyl alcohol and ethyl sulfide, or methyl alcohol and propyl sulfide are solvents for cellulose nitrate. Ethyl sulfoxide, an oxidation product of ethyl sulfide, has been suggested as a plasticizer for pyroxylin.

## SULFONATES

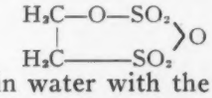
In the refining of mineral oils, particularly in the production of white pharmaceutical petrolatum (vaseline oils), with sulfuric acid, a certain amount of sulfonation occurs. Most lubricating oils contain reactive hydrocarbons which are partly removed from the oil by agitation with sulfuric acid. The sludges from such refining operations contain free acids, polymerized bodies, asphaltic materials, and oxidized substances together with various sulfonated products. The disposal of this acid sludge has long been a problem to the refiner. The acid is usually separated by the addition of water and reconcentrated for further treatment of distillates. The residual material is largely used as a fuel.

Numerous methods have been devised for the removal of the sulfonic acids from sludges and acid-treated oils. In general, separation is effected by some selective action such as that of solvents or adsorbents or by salting-out the acids in the form of their alkali or alkaline earth salts. As is evident, the sulfonic acids vary with the character of the sludge, the type of the original oil, and the refining treatment; hence the methods used for their recovery are generally adapted to a particular material.

Mineral-oil sulfonic acids, varieties of which are known as Kontakt, mahogany acids, and green acids, have found commercial interest. They have been used as detergents, reagents for hydrolizing fats to glycerol and acids, and textile reagents—i. e., wetting, penetrating, cleansing, or lubricating agents in textile treatment. On some types of dispersions, mineral-oil sulfonic acids act as stabilizers, but on others they exert a coagulating effect. In the petroleum industry the acids recovered from sludges have been used to prevent emulsification in subsequent refining operations. Various sulfonated mineral-oil fractions have been employed also in insecticide compositions. These applications have been so extensively investigated that to single out one as an illustration would be unfair to several hundred others.

The sulfonated mineral oils can be considered to consist largely of sulfonated paraffin and cycloparaffin (naphthene) hydrocarbons and to be derived from petroleum distillates. Aromatic sulfonic acids, though they may be formed also in small quantities in petroleum refining, are largely derived from coal tar. The petroleum sulfonic acids can thus be looked upon as by-products, whereas another series of compounds derived from olefins resulting from pyrolysis of petroleum distillates, for example, can be considered to be principal products.

When ethylene reacts with sulfur trioxide, a crystalline compound which melts at 80° C. (176° F.) is formed. This substance is carbyl sulfate:



It dissolves in water with the liberation of heat and the formation of ethionic acid, $HO_3S—C_2H_4—OSO_3H$, and isethionic acid, $HO_3S—C_2H_4—OH$. Salts of ester-like condensation products of ethionic acid and fatty acids—e. g., oleic acid, have appeared

as Igepons and have received much attention as wetting and cleansing agents, especially in textile treatment. Olefins resulting from the destructive oxidation of paraffin wax and naphthenic acids have also been sulfonated to give similar products.

Although petroleum sulfonic acids have been known and investigated for a long period, great technical interest was not manifested until recently with the development of products of superior detergent quality. The sulfation of olefins, resulting from the cracking of petroleum, has opened a wide field for the petroleum industry, and further developments quite as remarkable are already foreshadowed.

## NAPHTHENIC ACIDS

Other by-products from the refining of petroleum distillates are naphthenic acids, which are extracted from distillates by the alkali wash required in refining operations. The acids are obtained in the free state by acidifying the alkali with sulfuric acid or with sulfur dioxide. Their name is indicative of the fact that they were first observed in naphthene-containing crude oils. Later investigations of the structures and properties of naphthenic acids have shown also that they bear a chemical relation to the naphthene hydrocarbons, particularly to cyclopentane.

An interesting application of the heavy metal salts of these acids, the cobalt, lead, and manganese salts especially, is as paint and varnish driers. Solutions of such salts in hydrocarbons are known as Soligen driers. Linseed oil varnishes, containing 0.14 to 0.29 percent cobalt naphthenate, dry in 1 to 10 percent less time than those containing a resinate drier.

The sodium salts have been suggested as soaps. Their detergent action and lathering power are inferior to those of sodium palmitate or toilet soaps of mixed fatty acids however. A 40 percent solution of such soaps can be cooled to 0° C. (32° F.) for 30 minutes without effecting complete solidification, which indicates their possible use in the preparation of neutral soft and liquid soaps. Much larger quantities of sodium chloride are required to salt-out sodium naphthenates than coconut oil soaps from aqueous solutions.

## HYDROGEN

According to well-known processes, hydrocarbons and steam react at high temperatures in the presence of catalysts to give hydrogen and oxides of carbon. With methane this may be represented by:

$$CH_4 + H_2O \rightarrow CO + 3H_2$$
$$CH_4 + 2H_2O \rightarrow CO_2 + 4H_2$$

and for higher hydrocarbons by:

$$C_nH_{2n+2} + nH_2O \rightarrow nCO + (2n+1)H_2$$

These reactions are reversible, the equilibrium depending upon the temperatures and pressures employed. From an industrial point of view the chief interest in them lies in their application to the production of hydrogen from gaseous hydrocarbons, as more hydrogen can be obtained from a given quantity of methane, or other hydrocarbons, in this manner than by purely thermal decomposition. For example, the thermal decomposition of one volume of methane yields two volumes of hydrogen,

$$CH_4 \rightarrow C + 2H_2$$

but according to the preceding reaction it is possible to obtain as high as four volumes of hydrogen from one volume of methane.

Application of the above reactions is illustrated in the preparation of hydrogen from cracking gas and steam by a two-stage process.

In the first stage, reaction between the gases takes place at about 870° C. (1600° F.). The resulting mixture of hydrogen and carbon monoxide is further mixed with steam and conducted over a catalyst at about 455° C. (850° F.). By this second step an additional supply of hydrogen is obtained by the conversion of carbon monoxide into carbon dioxide according to the well-known water gas reaction:

$$CO + H_2O \rightarrow CO_2 + H_2$$

At the end of these two stages the gaseous mixture contains about 79 percent hydrogen, 20 percent carbon dioxide, and 1 percent of other gases. Carbon dioxide is removed by compressing the gases to a pressure of 240 pounds per square inch, followed by washing with water and then with aqueous triethanolamine. The final product contains more than 97 percent hydrogen and can be used in the hydrogenation of oils.

The hydrocarbon gases should be free of hydrogen sulfide, or other sulfur compounds, which poison the catalysts. The latter often consist of oxides of nickel, cobalt, iron, or aluminum, to which may be added activators, such as the oxides of chromium, vanadium, or magnesium.

## NATURAL GAS

Natural gas, which may be regarded as gaseous petroleum, consists primarily of methane, together with some ethane and propane, small quantities of butane, pentane, and other paraffins up to octane, and smaller proportions of carbon dioxide, hydrogen sulfide, and (in exceptional instances) helium. Natural gases are roughly divided into two classes, "wet" and "dry" gases, the former containing a large proportion of easily liquefiable hydrocarbons. Removal of these higher paraffins from natural gas by (*a*) compression, (*b*) absorption in oil, or (*c*) adsorption—e. g., with charcoal—yields natural-gas gasoline. Before blending this product with other motor fuels, it is subjected to stabilization or distillation under pressure, by which propane and some butanes are obtained. Natural gas is utilized primarily for the large-scale production of carbon black.

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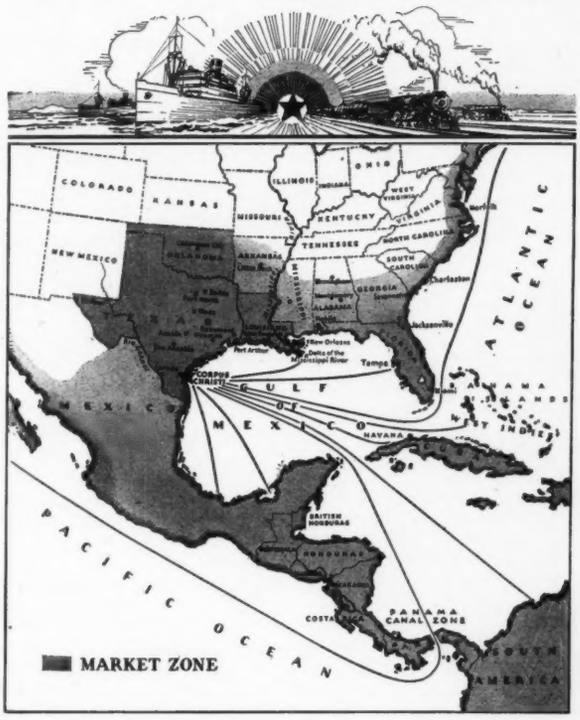


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*The abstracts here presented are selected from the current literature of science and technology to afford reference to fundamental information not easily available to all readers. Abstracts of articles appearing in readily obtainable trade journals are not included.*

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## Chemical Composition and Reactions

**Chemistry of Petroleum.** G. R. NIXON. *Jour. Inst. Pet. Tech.* **20** (1934) pp. 484-508.

A review of the work on the chemistry of petroleum during the year 1933. The chemistry of crude petroleum is first considered, and following that, that of gasoline, gas oil, lubricating oils, and asphalts. Work on the individual hydrocarbons and their physical and chemical properties is next considered. Polymerization, oxidation, hydrogenation, cracking, and combustion are reviewed. The chemistry of sulphur compounds is briefly considered, as well as that of various miscellaneous substances or products. A bibliography of two hundred and six references is included.

**Irritation of the Skin by Hydrocarbons.** WOLFGANG HEUBNER. *Arch. Pharm.* **272** (1934) pp. 379-83.

Experiments in which man was used as a subject showed that the irritating properties of paraffin hydrocarbons increases from pentane to heptane, but decreases from octane to decane. Dodecane was found to be practically inactive. The effects of cyclic hydrocarbons were also studied. It was concluded that the strongest skin irritants are those substances least reactive chemically and least soluble in water.

**Pyrolysis Studies.** C. D. HURD AND L. K. EILERS. *Ind. Eng. Chem.* **26** (1934) pp. 776-80.

In previous studies in the senior author's laboratory the pyrolysis of isobutylene was studied, and such matters as the influence of the size of the reaction tube, the contact time, the polymerization process, and the effect of metal tubes were not developed. These are discussed in the present paper. It was found that Ascoloy is non-catalytic. Nickel is slightly catalytic, iron more so, and Monel metal extraordinarily catalytic. The catalytic effect of the metal of the tube is evidenced by the lower decomposition temperature, the tendency of the reaction towards de-hydrogenation and carbonization. Using Ascoloy tubing, the following series of descending stabilities was obtained: ethylene, propylene, isobutylene, and 2-pentene. The liquid products from isobutylene at 700°C. or above contain both unsaturated and aromatic hydrocarbons. These were separated by a sulphuric-boric acid reagent. A study of diisobutylene shows that it need not be regarded as an essential intermediate in the pyrolytic polymerization of isobutylene. Theoretical considerations, confirmed by experimental data, are offered to show the necessity for caution in interpreting the results from the zero conversion method of determining initial products. Compounds that appear to persist at zero conversion may be secondary products as well as primary. Experiments with isobutylene in large and small tubes demonstrate that the extent of decomposition and the nature of the products formed are comparable if only temperature and time of contact in each are comparable. A bibliography of 18 references is included.

**The Thermal Decomposition of Cyclopentane and Methyl-cyclopentane.** B. A. KASANSKY AND A. F. PLANTE. *Berichte der deutschen chemischen Gesellschaft* **66** (1934) p. 1023.

The separation of the cyclopentane and the methyl cylopentane used is described. Quartz tubes 16 mm. in diameter were used for the pyrolysis. When cyclopentane was cracked at 650° unsaturated compounds formed 70 percent of the entire gas. At 730°C. the percent dropped to 55.8. Diolefins, including, probably, cyclopentadiene vapors, were approximately 1 percent. Paraffin hydrocarbons varied in amount. The use of a shorter time factor gave similar results. The pyrolysis of methyl cyclopentane gave more propylene than ethylene, notwithstanding the greater instability of ethylene. The gas contained iso-butylene and more paraffin than from cyclopentane. Cyclohexane formed more diolefins, principally butadiene, less propylene and more saturated hydrocarbons and hydrogen than cyclopentane. Liquid hydrocarbon products were obtained in such small quantities that it was difficult to investigate their nature.

## Fundamental Physical Data

**Contribution to the Problem of Heat Transfer in Turbulent Flow.** H. LORENZ. *Z. tech. Physik* **15** (1934) pp. 155-63, 201-6.

Existing data concerning the influence of temperature gradient, tube length and surface condition on heat transfer from a fluid to a tube wall are correlated. A graphical method for comparing heat transfer under different conditions is described.

**Conductivity-Temperature Curves of Pariffin Wax.** W. JACKSON. *Nature,* 133 (1934) pp. 647-8.

It was observed, peculiarly enough, that the conductivity of a sample of paraffin wax of 45-55°C. melting point, decreased with temperature and showed a hysteresis effect in the range of temperature 0-50°C. Conductivity measurements were made with direct current. The results of a series of measurements under a continuously applied voltage gradient of 500 volts per centimeter is shown graphically. The author believes that the phenomenon is probably connected with the presence in the wax at temperatures below the solidifying point of a solid crystalline network in which is interspersed a liquid phase. The boundary surface between the two phases would have a greater conductivity than either phase, and wherever continuous would form a highly conducting bridge between the electrodes.

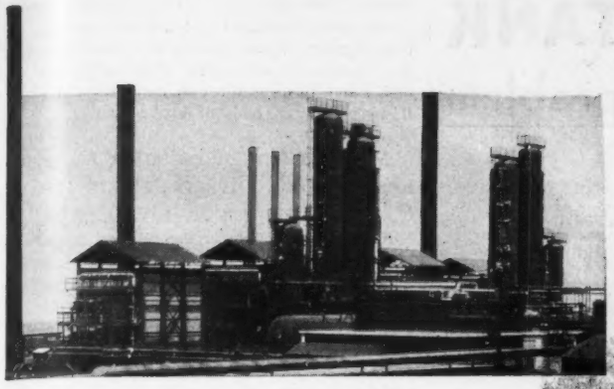
**Petroleum Literature.** W. S. E. CLARK. *Jour. Inst, Pet. Tech.* **20** (1934) pp. 636-50.

A review of the sources of information in the petroleum literature for 1933. This includes reference to annual reports of technical societies, publications of government bureaus or departments, as well as books. Reference is also made to particular articles when these are of general review nature and contain extensive bibliographies. A bibliography of one hundred and sixty-seven references is appended.

## Manufacture: Processes and Plant

**Heat Transfer Involving Turbulent Fluids. Correlation of Heating and Cooling Data.** W. A. KAYE AND C. C. FURNAS. *Ind. Eng. Chem.* **26** (1934) pp. 783-86.

It has been generally conceded that coefficients of heat transfer between turbulent fluids and a solid wall are different when the fluid is being heated and when it is being cooled. It is noted that the discrepancies between heating and cooling coefficients are in opposite directions for gases and liquids. The authors advance a theory to explain the difference between the film coefficients of heat transfer on heating and cooling fluids in turbulent flow in contact with solid surfaces. The relation between the two is a simple function of the main



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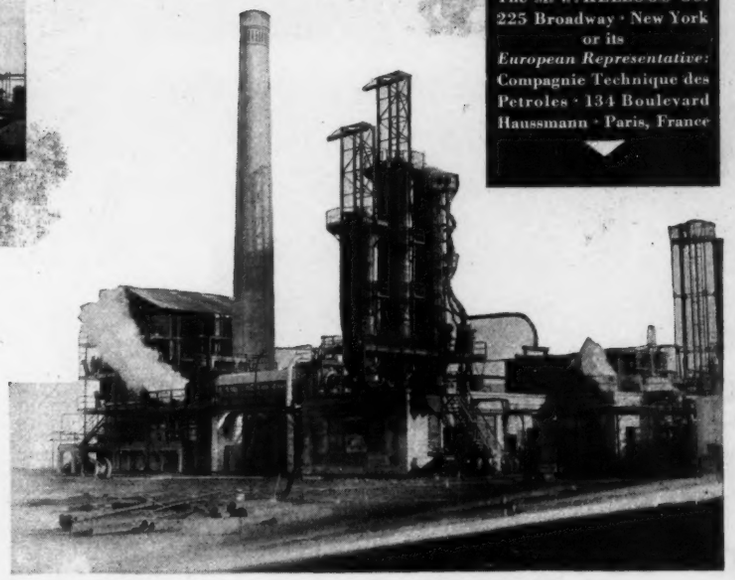

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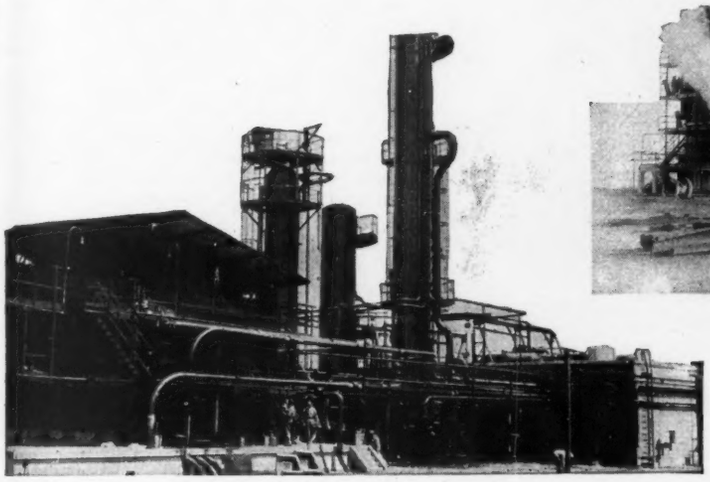

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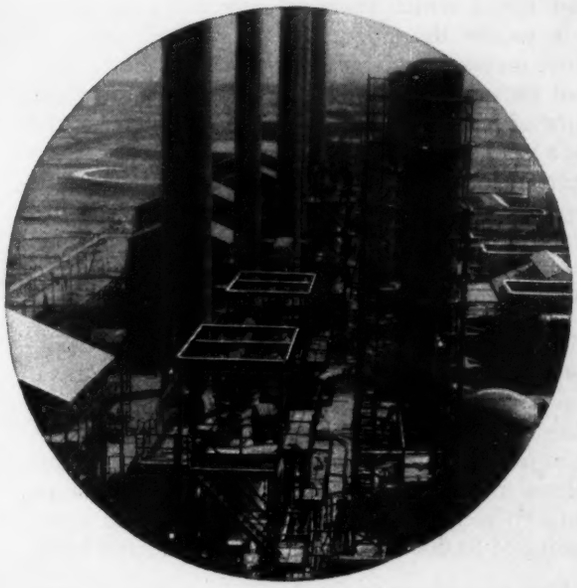

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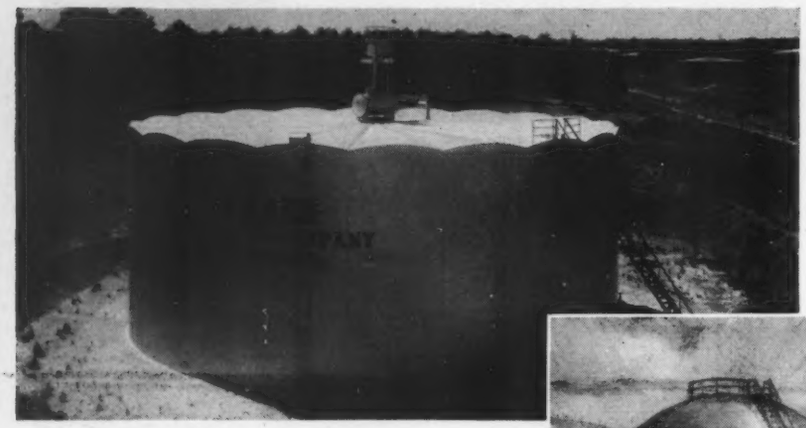

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stream viscosity. The work of various investigators was considered and it was found that the theory gives a satisfactory correlation between the heating and cooling coefficients for oils and water flowing through conduits and for air flowing through beds of broken solids.

**Measurement of radiation (in a gas-fired boiler).** WALTER BRUMMERHOFF. *Gas u. Wasserfach* **76**, (1933) pp. 929-33.

A study was made of the relative proportions of heat transferred by radiation and convection under a gas-fired boiler. The heat not radiated and contained in the gases from which it would be transferred convectively was removed by withdrawing the combustion products through insulated pipes at such a rate that the normal carbon dioxide content was maintained. The total heat with normal operation was also measured. The difference between the two was the heat transferred convectively. Since ordinary methods are not satisfactory for determining the temperature of the gases within the fire tubes, a new method was developed, depending on the measurement of gas velocity and the calculation of temperature by means of the equation $t = 273\ [p_d(3600\ F/Qf_v)^2\ (2g/\gamma)]\ -\ 273$, or $t = (Cp_d/Q^2f_v^2) - 273$, in which $p_d$ is the difference between static and dynamic pressure on a Prandtl Pitot tube in the fire tube measured in mm. water column, F is the internal cross section of the heating tube in sq. m., Q is the vol. of flue gas in cu. m. at 0° and 760 mm. Hg per hr, per cu. m. of fuel gas, $f_v$ is the calibration factor and $\gamma$ is the sp. gr. of the hot gas in kg, per cu. m. Measurements were taken for one tube in the outer ring, one in the central, and one in the inner group.

**Treatment of (removal of hydrogen sulphide etc. from) Mineral Oil Distillates.** R. H. CROSBY AND B. R. CARNEY. *Petroleum* **30** No. 11 (1934) pp. 5-6.

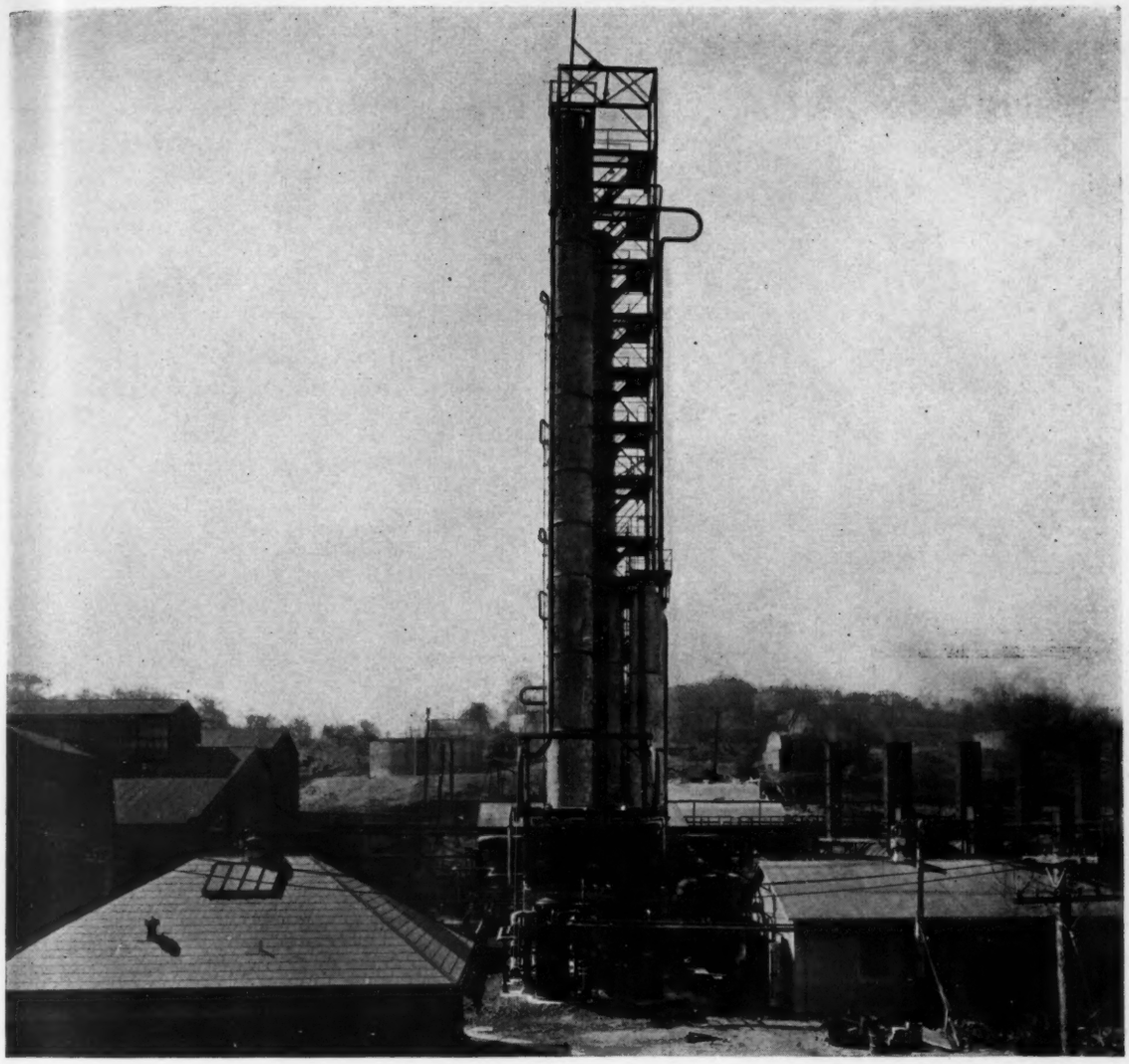
Acid impurities, including hydrogen sulphide, are removed from mineral oils by treatment with a 2-5% suspension of calcium hydroxide and ferric hydroxide under pressure of 1.35-2.3 atmospreres and in absence of air. Difficulty with emulsification is thus avoided. The oil is then treated with a light wash of aqueous caustic soda to remove calcium salts of naphthenic acids that may remain in solution.

**Transportation and Storage.** A. C. HARTLEY. *Jour. Inst. Pet. Tech.* **20** (1934) pp. 576-89.

A review of the work and published literature on transportation and storage during the year 1933. The materials for, and methods of, constructing pipe lines are first considered, including pumping stations and submarine pipe lines. Erosion and the maintenance of pipe lines is reviewed. Storage tankage, including particularly the steel balloon, is briefly discussed. Reference is made to methods of fire prevention and fire fighting. A bibliography of 13 references is included.

**Welding in Chemical Industries.** C. O. SANDSTROM. *Chem. & Met. Eng.* **41** (1934) pp. 360-64.

The author considers the position of welding as thoroughly established. He is convinced that the majority of weld failures have been caused by faulty design and not by poor welding. The materials that are to be welded are briefly discussed. Types of butt-welded joints are described and compared to riveted joints in the matter of eccentricity. Various types of butt-strap joints are considered and the errors in making them discussed. Fillet welds and the common and proper methods of making them are considered. End and edge welds, calking and plug welds, and the welding of sheet steel



# Compact Fractionation

To meet particular requirements in Pennsylvania, this installation includes —crude oil heater, fractionating tower, heat exchangers and coolers; lube oil heater, fractionating tower, heat exchangers and coolers; naphtha rerun unit of reboiler fractionating tower, external stripping tower, coolers and auxiliaries.

The installation is designed for great flexibility, compactness and low operating cost. The crude oil fractionation gives: gasoline, naphtha, kerosene, furnace oil, fuel oil or gas oil, pressable wax distillate, heavy wax distillate and cylinder stock. When cylinder stock solution is charged, the lube tower produces: naphtha overhead, two small side streams of gas oil and neutral oil with bright stock bottoms. The naphtha unit yields light gasoline, intermediate naphtha and heavy naphtha, from the bottom.

Provision is made for changing specifications of products without altering the tray arrangement.

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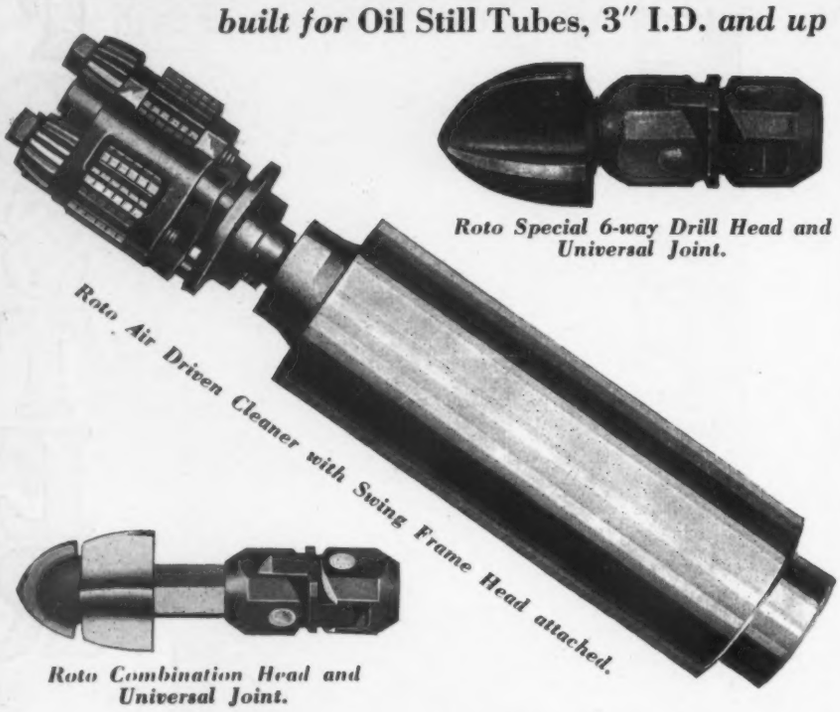

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## *of Liquid Levels in Refining*

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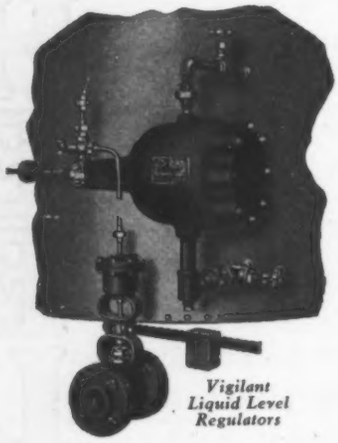

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are discussed. The designer of welds will find a great deal of helpful information in this article.

## Products: Properties and Utilization

**Special Products.** S. F. BIRCH. *Jour. Inst. Pet. Tech.* **20** (1934) pp. 453-6.

A review of special products from petroleum, including carbon black, olefins, alcohol, ethylene oxide, chlorinated paraffin, insecticides and spray fluids, and special bitumins and waxes. A bibliography of 37 references is given.

**Formation of Carbon Deposits in Internal Combustion Engines, Part I.** H. N. BASSETT. *Gas & Oil Power,* **29** (April, 1934) p. 110.

Although the flame temperature in an engine is high enough to burn lubricating oil completely, the time is insufficient and the result is partial combustion and distillation. Combustion is also hindered by the relatively low temperature of the cylinder walls. Semi-carbonized oil deposits on the piston head and in the ports. The nature and quantity of this carbon vary with the type of oil and the condition of use. Stability towards oxidation is an important factor in avoiding carbon formation and there is much to recommend use of antioxidants. The fact that an oil does not contain hard asphalt, as shown by the Institution of Petroleum Technologists test method, does not mean that it will not form carbon in use. Excessive treatment of the oil to remove asphalt may impair the lubricating properties of the oil. The important consideration is the proportion of components readily oxidized to asphaltic substances. In determining the carbon forming tendency of oils, the volatility or evaporation tests are useless. The I.P.T. oxidation test, although not wholly representative of working conditions, does indicate the resistance of an oil to oxidation. The Conradson and Ramsbottom carbon residue tests do not give comparable results and are of limited value in predicting carbon formation.

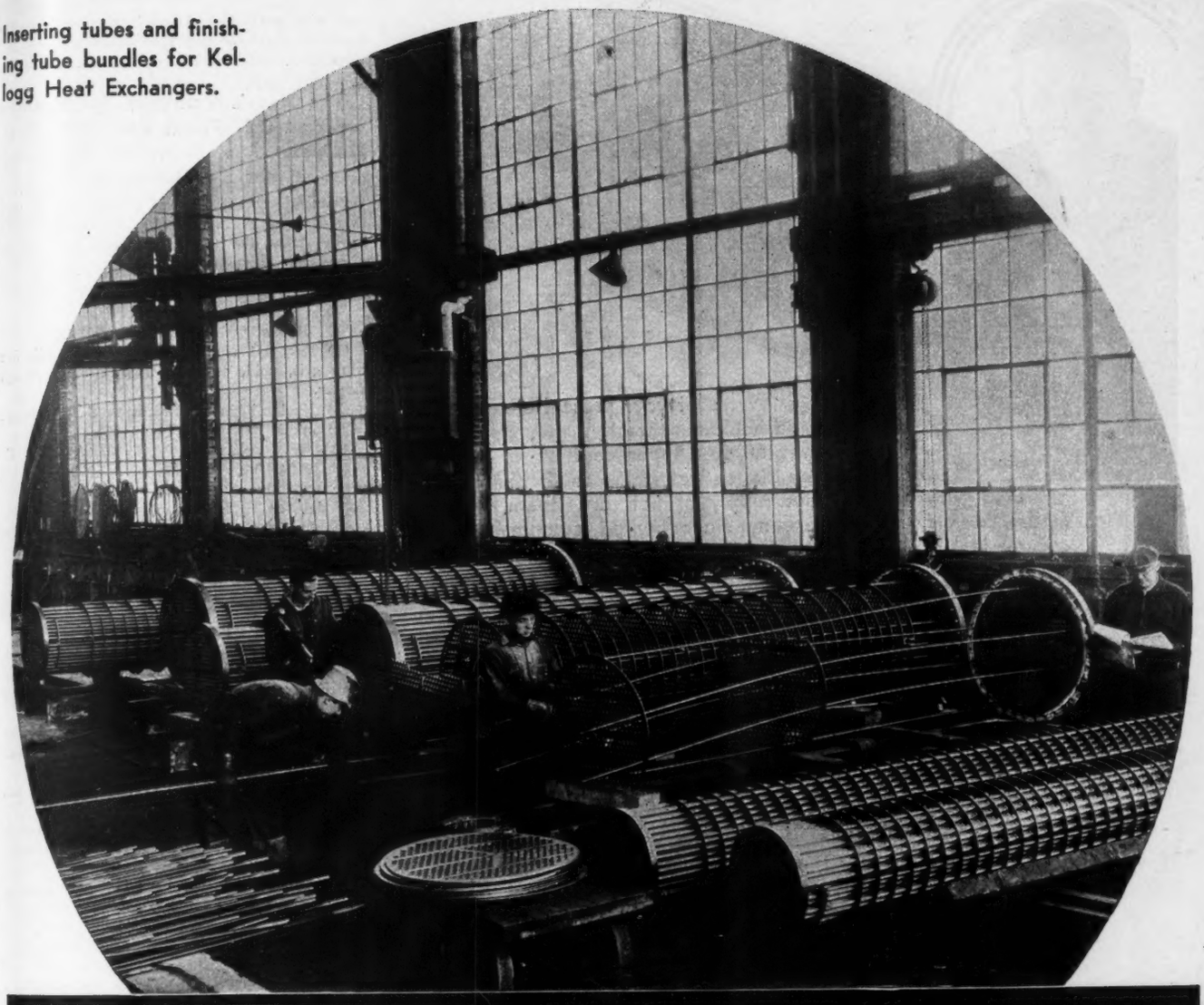
**Properties of Automobile Lubricants.** R. OREL. *Petr. Z.* **30** (1934) p. 15.

Dilution and its dependence on the oil and on operating conditions, sludge formation, the limits of use of the oil, and the value of the initial viscosity and carbon deposits were studied. Dilution occurred in three phases: first, after 400-500 kms. driving, a rapid fall in viscosity amounting to 50-70% of the initial kinematic viscosity at 25°C.; second, the oil becomes enriched with naphtha; and third, a form of solidification occurs. Viscosity decrease is at a maximum when momentary stiffening occurs. Sludge formation depends on working conditions and on the presence of water. It is less in the first period and increases as the oil is used further. Carbon deposits are caused by incomplete combustion of the sprayed oil, the half-burnt oil partly clinging to the pistons and cylinder walls. The same oil forms different quantities of carbon with different motors. Oil consumption does not depend upon the formation of carbon deposits.

**Causes and Effects of Sludge Formation in Motor Oils.** D. P. BARNARD, E. R. BARNARD, T. H. ROGERS, B. H. SHOEMAKER AND R. E. WILKIN. *S.A.E. Journal* **34** (1934) pp. 167-78, 181T.

The formation of asphaltines through oxidation is the primary cause of sludge formation in motor oil. The test involving the oxidation of 300 cc. of oil in a glass tube at 341°F. with 10 liters per hour of air for 50-200 hours,

Inserting tubes and finishing tube bundles for Kellogg Heat Exchangers.



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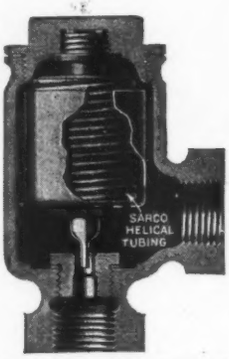

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and with periodical determination of naphtha-insoluble material and the viscosity, gives the correlation with engine performance of the oil.

**The Navy's Tentative Specifications for Fuel Oil—Fiscal Year 1935.** F. M. McGEARY. *Jour. Amer. Soc. Naval Engineers* **46**, No. 2 (1934) p. 212.

Changing conditions, and particularly the fact that Bunker A fuel oil has practically disappeared from the market, require the drawing of a new specification for a compromise fuel oil for the U. S. Navy. The old and new specifications follow:

PREVIOUS SPECIFICATION

(a) Fuel oil shall be hydrocarbon oil, free from grit, acid, and fibrous and other foreign matter.

(b) Fuel oil shall conform to the following:

| Bunker | A | B | C |
|---|---|---|---|
| Flash point, degrees F. minimum | 150 | 150 | 150 |
| Viscosity, Saybolt Furol, secs. at | | | |
| 77 degrees F. maximum ..... | 100 | ... | ... |
| at 122 degrees F. maximum. ... | | 100 | 300 |
| Water and sediment, combined, | | | |
| percent maximum ..... | 1 | 1 | ... |
| Water, percent maximum ..... ... | | ... | ... |
| Sediment, percent maximum.... ... | | ... | 0.25 |

PRESENT SPECIFICATIONS

(a) Navy fuel oil shall be a hydrocarbon oil, free from grit, acid, and fibrous or other foreign matters likely to clog or injure the burners or valves.

(b) The flash point shall not be lower than 150 degrees F. when tested in a Pensky-Martens closed tester.

(c) The viscosity shall be measured at two points as follows:

(1) The viscosity in Saybolt Furol seconds shall not be greater than 750 seconds at 77 degrees F.

(2) The temperature at which the viscosity in Saybolt Universal seconds is 150 shall be not greater than 185 degrees F. The oil as delivered shall not contain more than 1.0 percent of total water. For deliveries by supplier's tankers or barge, oil containing not more than 2.0 percent of total water is acceptable with a reduction in price for all water in excess of 1.0 percent.

Note—The reduction shall be (1.25 times excess water percent) percent of contract price.

(d) Ash shall not be greater than .12 percent.

(e) The oil shall have a fire point of not less than 220 degrees F. If water content of the oil prevents an accurate determination of fire point, the oil will be acceptable if the foaming due to water starts below the fire point and at a temperature not lower than 195 degrees F. If the flash point is 175 degrees F. or higher the fire point need not be determined.

(f) The oil shall be free from a tendency to deposit excessively in fuel oil heaters when being heated for atomization at a viscosity of 150 S.S.U., for all rates of flow from at anchor to full power, with sudden changes of rate of flow and with steam to oil heater manually controlled.

(g) The oil shall be free from a tendency to sludge excessively in storage at temperatures from 40 degrees F. to 100 degrees F.

It will be seen that ash, heater-fouling tendency, and stability in storage are the subject of new requirements, and that nothing explicit is given under (f) and (g) as to the method of making the tests.

**Analysis and Testing.** C. CHILVERS. *Jour. Inst. Pet. Tech.* **20** (1934) pp. 457-83.

A review of the work during 1933 on analysis

Code or no code, competing refiners will take your customers away from you if they are making better gasoline than you are

Nobody takes customers away from a Dubbs refiner He wins new customers instead of losing old ones because they like Dubbscracked gasoline

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and testing. Work of the World Petroleum Congress, as well as of national standard associations, is briefly discussed. Developments of analytical procedures for application to crude petroleum, gas, motor fuels, sulphur determination, kerosene, gas oil, lubricants, insulating oils, viscometry, bituminous materials and special products are covered in detail. Developments in the determination of physical properties in laboratory distillation and in the making of chemcal tests are reviewed. A bibliography of 322 references is appended.

**Viscosity of Petroleum Products. Methods and Apparatus for Viscosity Measurement.** E. A. WILLIHNGANZ, W. B. MCCLUER, M. R. FENSKE AND R. V. MCGREW. *Ind. Eng. Chem., Anal, Ed.,* **6** (1934) pp. 231-4.

The probable sources of error encountered in viscometry practice are discussed and methods for eliminating or minimizing these effects are given. Modified Ostwald pipets designed to minimize the probable sources of error have been constructed, many of which have been satisfactory in use for two years. The pipets are of rugged construction and the loss due to breakage is negligible. Viscosity determinations made by various research workers using different apparatus normally check within 0.2 percent. The pipets require only 5 cc. of liquid for viscosity determinations. Their application to petroleum work and to research work in general is indicated.


# HOW this Midwest Refinery Cut Costs Reduced Fire Hazards Increased Flexibility ....with G-E Gear-Motors

*Midwest refinery, showing 16 of the 24 G-E gear-motors. Arrows point to G-E explosion-proof magnetic switches. Note the motor shown below with cover removed*

By installing 24 G-E 5-hp. gear-motors to drive equipment previously operated by three 50-hp. motors, a large midwest oil refinery has . . .

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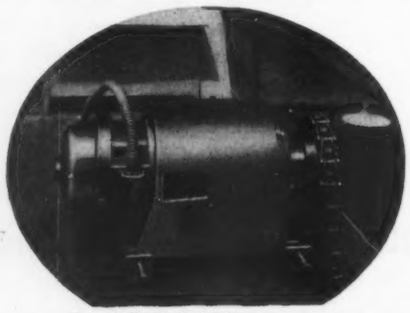


*One of the 24 G-E 5-hp. gear-motors*

020-87

GENERAL ELECTRIC


**The Viscosity of Pitch.** A. B. MANNING. *Fuel Research Technical Paper No. 39, Dept. of Scientific and Industrial Research, London,* 1933.

Three methods were used for determining the viscosity of pitch over the temperature range from 30 to 110°C. Between 30 and 40°C. the rate of flow of a disc of the pitch under pressure was measured. Between 30 and 80°C. a rotating-cylinder viscometer was adopted. Above 80°C. measurements of viscosity were made by measuring the time required for the pitch to rise in a vacuum from one mark to another on the viscometer tube. Tubes of fused silica were used because they were more durable than glass. It was concluded that the coal-tar pitches are true viscous liquids and have a coefficient of viscosity that is independent of the rate of shear and depends only upon temperature. Bitumen, or petroleum pitch, is unlike coal-tar pitch in that its viscosity decreases with increase in the rate of shear to which it is subjected. Bitumen is regarded as having a structure similar to that of a gel. The temperature coefficient of viscosity of bitumen is much less than that of coal-tar pitches for the same rate of shear. An appropriate temperature for the determination of the viscosity of a pitch is the briquetting temperature, as for example, 95°C.

**Determination of the Colour of Solid Paraffin and Its Resistance to the Action of Light.** D. BARGMAN. *Rev. Petr., No.* 570 (March 17, 1934) pp. 295-296.

The reversion of color of sample of paraffin wax caused by the action of light may be occasioned by the oil content or by the degree of refining. Other workers have obtained a light stable wax by removing the oil from a reverted paraffin through recrystallization from ethylene dischloride. Schille showed that the resistance of paraffin to light varies inversely with the oil content. Ultra-violet light from a mercury-vapor lamp has been found more active than sunlight. Bomberg proposed a method for the determination of the color of solid paraffin that comprised the comparison of colors of a plaque of paraffin 1 cm. thick and a column of potassium dichromate solution. Difficulty is encountered because of the difference in shade between paraffin and the standard, and also on account of the mottles of the paraffin. The paraffin can be freed of mottles and air bubbles by melting and transferring to a vacuum desiccator for slow cooling to room temperature. The results of a series of experiments showed that one hour's exposure to ultra-violet rays was equivalent to eight days of sunlight in the matter of change of color of paraffin wax. Tables are given showing the colors of samples of solid paraffin of different oil content and penetration before and after exposure to ultra-violet light.

**Influence of Refining on the Oxidizability and the Oiliness of Lubricating Oils.** N. I. CHERNOZHUKOV AND A. M. GUTZAIT. *Repts. Lubricating Oil Comm. U.S.S.R.* **3** (1933) pp. 1-23.

Light spindle oils, machine oil distillates and bottoms from Baku and Emba crude oils were studied. The oxidizability was tested by the Butkov method and the oiliness by the Dallwitz-Wegener method. Fuller's earth treatment was effected by shaking the oil at 50°C. with fuller's earth for 30 minutes. Emba spindle oil distillate showed a continuous increase in oiliness with treatment, a maximum being reached with 15% fuller's earth. The Baku distillate of corresponding grade lost its oiliness when treated with large proportions of fuller's earth. It was found that oiliness had no relation to acid con-

tent. The color of Emba distillate improved in direct ratio to the amount of fuller's earth used in treatment, while the color of Baku oil in relation to quantity of earth is expressed as a curve. Neither oil is of stable color when exposed to the light for some time. The acidity of the Emba distillate improved in direct proportion to the quantity of fuller's earth used in treatment, but the relationship of Baku distillate is expressed by a curve. The Baku distillate yields a stable oil if treated with 5% of fuller's earth. However, 30-40% of fuller's earth was required to stabilize or remove the resins from Emba distillate. This is explained on the basis that Emba oil when oxidized yields asphaltic products, while Baku oil yields only hydroxy acids. Baku spindle oil distillate, after treatment with fuller's earth at 150°C., lost its stability to oxidation and color. Emba distillate, however, yielded a stable oil after treatment with only 5% of fuller's earth.

**Lubrication of Bearings.** H. BRILLIE. *Shipbuilding and Shipping Record,* 43 (1934) pp. 336-338.

The Warlop system of oil circuit-bearing is described. In this bearing the intermediate bushing between the shaft and the plummer-block is sub-divided into a large number of sections by a series of transverse planes. A series of holes at regular distance on the circumference corresponds to each of these sections, and, on the other hand, grooves outside the bushing correspond to the holes. The holes are stepped from one section to the next. A test with thick grease, instead of fixing the bushing, left it free. The shaft was revolved at approximately 2000 r.p.m. It ran smoothly and regularly and with no vibration. After about 20 seconds the bushing started revolving regularly at the rate of 1 r.p.m. in a direction opposite to that of the shaft. This was caused by the driving effect of return currents of lubricant films. It was found that the Warlop bushing gives frictional coefficients distinctly below those for ball bearings when used with oils of different viscosities and also with grease.

**Origin and Development of the Heavy Oil Engine.** S. HICKS. *Trans. Inst. Mar. Eng.* 46 (1934) pp. 42-63.

An abstract of the Third Ackroyd-Stuart Award Paper in 1932-3, covering the development of all kinds of heavy oil engines including stationary, marine, automobile and aero from the time of the Ackroyd-Stuart patents of 1890 to the present time. The article is comprehensive and profusely illustrated.

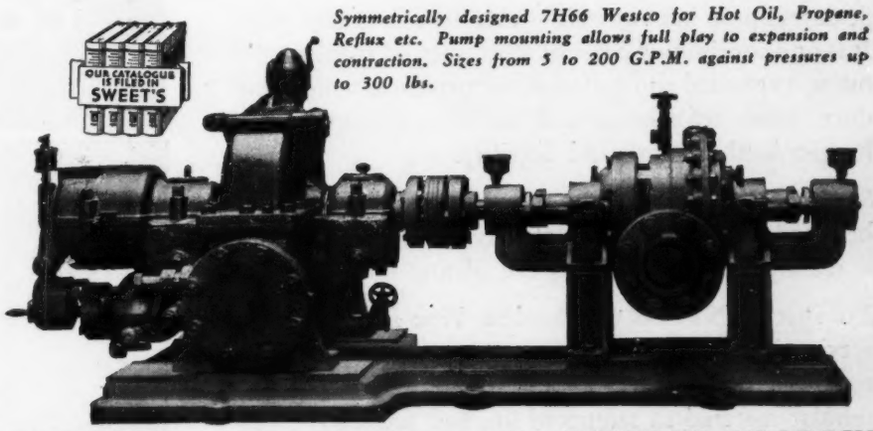
## Natural Gas and Natural Gasoline

**Analysis of Gaseous Hydrocarbons. A Method for Determining Gaseous Paraffins and Olefins.** H. TROPSCH AND W. J. MATTOX. *Ind. Eng. Chem., Anal. Ed.,* 6 (1934) pp. 235-41.

In anther publication the authors have reviewed the subject of gaseous hydrocarbon mixtures analysis. This study showed that specific tests are not generally available for the direct determination of individual hydrocarbons in complex mixtures, and that resort must be made to fractional distillation analysis rather than to difference in chemical behavior. In this article a method of hydrocarbon analysis is described in which, by fractional condensation, small samples (500 cc. or less) of complex mixtures are separated into composite fractions containing both two and three, and both three and four carbon atoms per molecule. Density determinations of these composite fractions containing paraffins and olefins and the same fractions after the removal of the olefins, by means of a Stock electromagnetic gas-density balance, give data from which the percentage of each hydrocarbon is readily derived. Pentane and hydrocarbons of higher molecular weight are determined in the same fractionation. The accuracy of the method has been verified by the analysis of carefully prepared synthetic mixtures. The method has been applied to various types of refinery gases. The procedure is rapid and the cost is low.

**Application of Refrigeration to the Manufacture of Natural Gasoline.** F. L. KALLAM. *Chem. & Met. Eng.* 41 (1934) pp. 355-7.

The author calls attention to the fact that the economic advantage of employing refrigeration in the manufacture of natural gasoline is usually dismissed without consideration. However, a decrease in lean oil temperature of 1°F. represents an increase in absorption efficiency of 2%. Furthermore, the lower the temperature in the absorber, the more selective the action of the oil, for the reason that the quantity of any component absorbed is inversely proportional to its vapor pressure, and the vapor pressure of the heavier and more desirable components rises faster than that of the lighter fractions. Types of refrigeration machines available and those most suitable for use in the natural gasoline industry are discussed.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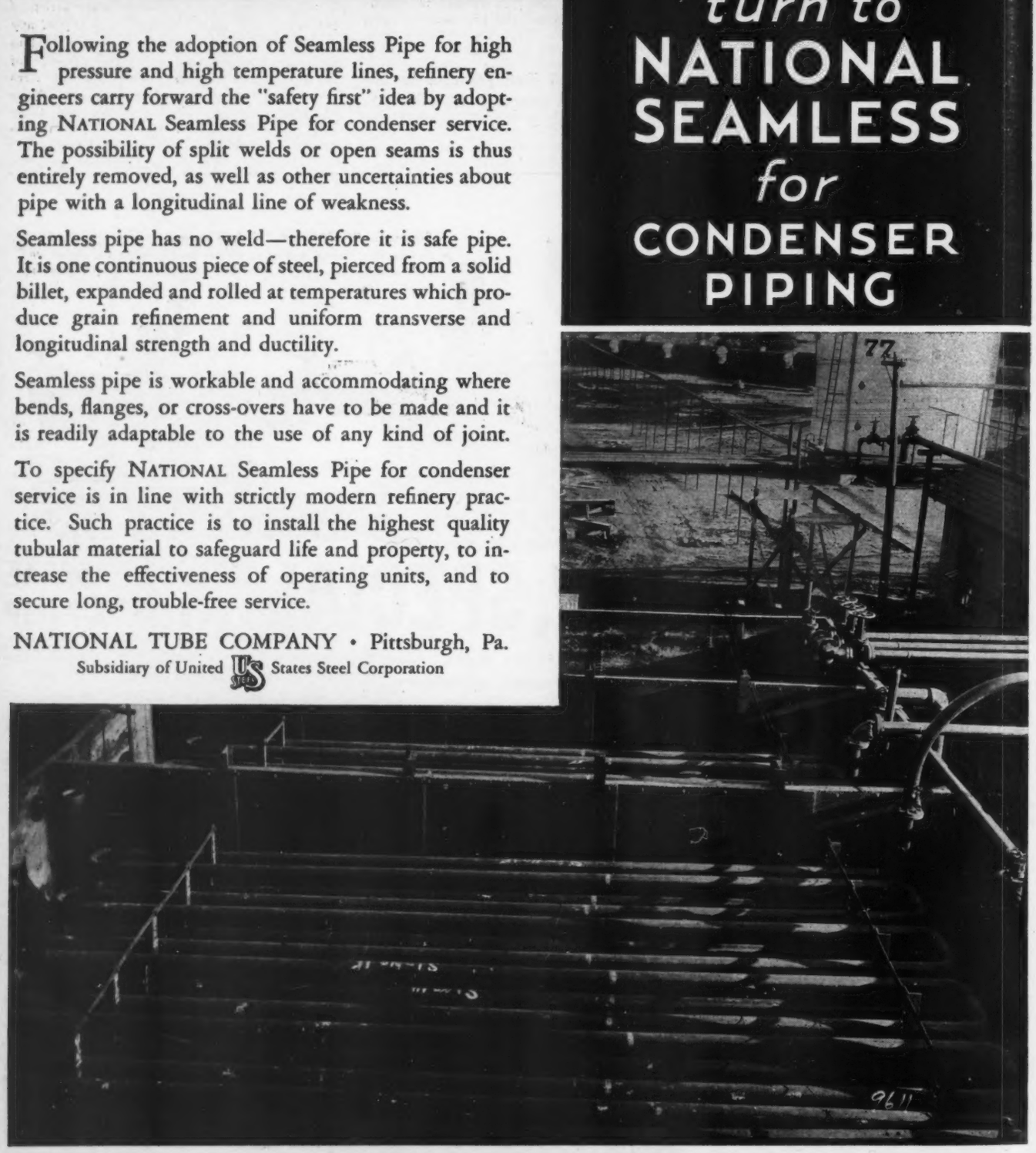

# NATIONAL SEAMLESS

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# THE MONTH IN REVIEW

## S-N Tri-Process Of Dewaxing

AT the recent inauguration of the new Standard Franco-Americaine de Raffinage plant at Port-Jerome—the largest refinery in France—the Separator-Nobel dewaxing plant attracted attention. It is the first large commercial plant of its kind put into operation, and the results obtained in actual service have surpassed guarantees.

The Separator-Nobel dewaxing process uses trichlorethylene as a solvent, and, in consequence, the centrifugal separators discharge the wax as the lighter and the "oil-tri" blend as the heavier component. The efficiency and economy in operation thus obtained are illustrated by the following figures from Port-Jerome:

| Type of oil | Motor oil I | Motor oil II | Cylinder oil |
|---|---|---|---|
| Dilution, oil: tri by vol. | 54 : 46 | 52.5 : 47.5 | 54 : 46 |
| Chilling temperature | —25°C.(—13°F) | —25°C.(—13°F) | —22°C.(—8°F) |
| Throughput of each centrifuge, dewaxed, clay-finished oil, lit/hour | 540 | 675 | 700 |
| bbls/24 hours | 81 | 100 | 105 |
| Pour point of dewaxed oil | —21°C.(— 6°F) | —11°C.(—12°F) | — 9°C.(16°F) |
| Cloud point of dewaxed oil | —19°C.(— 2°F) | — 8°C.(17.5°F) | Not determined |
| Yield of finished oil, % by vol. | 95 | 95.5 | 97.2 |

The comparatively small difference between chilling temperature and pour point of the first motor oil is noteworthy and also the slight difference between pour point and cloud point.

The above results were obtained during continuous runs for several days and nights, and since they exceeded the guarantees with an ample margin, the plant was definitely accepted and has by now been in successful continuous operation for more than two months.

It is interesting to note that although the wax is of typical crystalline structure, it is easily and continuously discharged from the centrifugal bowl, the machines having been run continuously without cleaning of the bowls, until each batch of oil has been finished, i.e., up to 150 hours without interruption.

This feature is said to be largely due to the use of trichlorethylene as a diluent, seeing that the wax is discharged from the center of the centrifugal bowl as the specific lighter component, as already mentioned.

Besides six De Laval S-N centrifugal separators, the dewaxing plant comprises proportioning and mixing equipment for oil and "tri," refrigerating plant with continuous chillers and distilling equipment for recovering the "tri" from the oil and wax. The whole system is hermetically closed and the losses of "tri" are limited to a fraction of a percent of the quantity of oil treated.

The refrigerating plant has a capacity of about 120,000 kgcal/hour, which has proved amply sufficient for the six separators, the total output of which amounts to 500-630 barrels of dewaxed oil per day. The operating costs per unit of finished oil are thus low, which is due to the comparatively low degree of dilution and rapid chilling characterizing the S-N tri-method of dewaxing.

## A. I. Ch. E. Announces 1934 Meeting Plans

AMERICAN Institute of Chemical Engineers has announced that its 1934 annual meeting will be held in Pittsburgh, November 15, 16 and 17. On Wednesday evening, November 14, there will be a meeting of the institute council. The Hotel Schenley has been selected as convention headquarters since it is located in the heart of the Oakland district, within easy access of the Mellon Institute, the University of Pittsburgh, the Carnegie Institute of Technology and Phipps Conservatory.

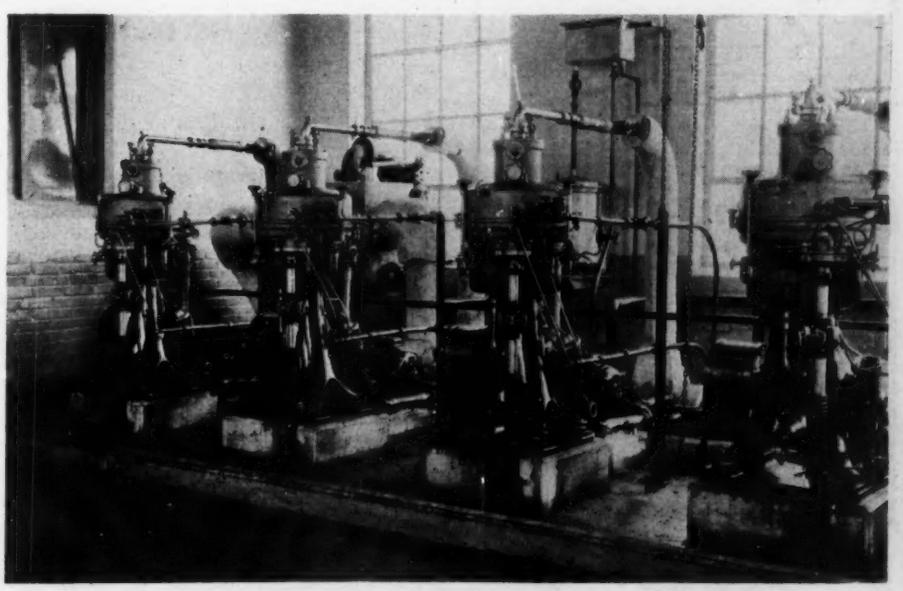
W. N. Jones, director of the College of Engineering, Carnegie Institute of Technology, is chairman of the committee on arrangements. A. G. Peterkin and J. H. James and the local committee on papers promise an interesting and instructive technical session. The industrial plants of the district are responding graciously to requests for plant visits. The new building which will house the Mellon Institute of Industrial Research will be inspected. On Saturday, November 17, the football teams of Duquesne University and Carnegie Institute of Technology will play at Pitt Stadium.

## Effect of Temperature On Properties of Metals

THE A.S.M.E.-A.S.T.M. (American Society of Mechanical Engineers and American Society for Testing Materials), Joint Research Committee on Effect of Temperature on the Properties of Metals, at its last meeting, voted to draw up a definite plan, for submission by letter ballot, for experimental study of methods of evaluating the load carrying abilities of metals under prolonged stress and high temperatures.

The "creep" of selected materials will be studied by test runs to be carried out, not merely for 1000 or 2000 hours, but for three years, or say 25,000 hours, in order that the validity of various methods of approximation and extrapolation and the relation of various "short-cut" methods to actual long-time performance may be evaluated.

It also voted to study experimentally the progressive changes in toughness, dur-



***Dewaxing plant at Port-Jerome, France.***

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ing long exposure to high temperatures, of the 18-8 stainless steel whose creep properties have already been studied in detail by the committee and which are being reported in the July "Transactions" of the A.S.M.E. This continuation of the prior work on this steel is aimed to throw light upon the fact that this lot of material, though free from added "stabilizing" elements, was not embrittled by long sojourn under load at high temperatures in the creep tests. The question whether this austenitic steel passed through a brittle stage, without evidence in the creep curves of any change in load-carrying ability, and then recovered from its brittle state, or whether it never did become embrittled, is of both theoretical and industrial importance, and a material whose other properties have been so thoroughly studied as is the case with this steel, is especially suited for such a study.

Besides these projects, which are in line with its past activities, the committee is extending its fields of active interest to include other matters of pressing industrial and engineering importance. The subcommittee on oil refinery problems is working out an acceptable plan for procurement of experimental and operating information on still tubes for the oil-cracking industry. Petroleum refinery engineers, tube makers, and the joint committee are cooperating in the effort to apply the most modern methods of evaluation of properties and performance toward the development of better tubes and their most economical engineering application.

Another activity, also with a bearing on the oil industry, in relation to dewaxing problems, but also with many other industrial bearings, is the correlation of data on properties of metals at sub-zero temperatures. Many of the members of the Joint Committee are engaged in getting new data in this relatively unexplored field, and the formation of a new sub-committee to deal particularly with this problem will serve to unify these data and make them more generally available.

Still another new activity is a similar correlation of data on seizure—i.e., the effect of temperature on metal-to-metal wear, galling and sticking. Users and makers of valves that operate at elevated temperatures find this an important problem in which available information is chiefly of an empirical nature. It is hoped to aid in the transition from empiricism toward a more sound basis for engineering design and selection of materials.

Thus the joint committee, while rigidly adhering to its policy of continued clarification of the fundamentals, is showing itself responsive to the engineering needs of the day in its selection of fields for immediate and intense activity.

## Designed by Burrell-Mase

In the article "Recovery and Stabilization Systems Combined at Panhandle Plant," page 289 of the August, 1934, issue of THE REFINER, describing a novel system of recovery and stabilization at the refinery of Panhandle Refining Company, Wichita Falls, Texas, information was omitted to the effect that the system was designed by Burrell-Mase Engineering Company, Pittsburgh and Tulsa. The installation is similar to that operated by Bell Oil & Gas Company, at Grandfield, Oklahoma, which was described in detail under the title "Combined Vapor Recovery and Stabilization System," on page 157 of the May, 1934, issue of THE REFINER. This latter installation was also designed by Burrell-Mase Engineering Company. In calling attention to the omission, Col. G. A. Burrell states that the new pressure distillate stabilizer and vapor recovery unit is the subject of a patent application; the patent not being for the purpose of exacting royalties but for infringement protection.

## Announce September Gasoline Allocation

ALLOCATION of allowable gasoline production for September, as announced by the planning and coordination committee, will be as follows:

East Coast district, 5,400,000 barrels; Appalachian, 1,375,000; Indiana-Illinois-Kentucky, 5,250,000; Oklahoma-Kansas-Missouri, 4,100,000; Inland Texas, 2,400,000; Texas Gulf Coast, 6,000,000; Louisiana Gulf Coast, 1,100,000; North Louisiana-Arkansas, 760,000; Rocky Mountain, 715,000; California 4,700,000; held in reserve for board of review; 580,000 barrels.

## Grisell Goes With Kendall

T. O. GRISELL, for the past two years executive secretary of the Pennsylvania Grade Crude Oil Association, has resigned to become sales manager for Kendall Refining Company, Bradford, Pennsylvania. He takes the position recently made vacant by the resignation of I. H. Shearer and will assume his new duties on October 1. It was in a great measure through Grisell's efforts that the work of the association with which he has been connected was brought to such a high plane as to command international recognition in the industry. Under his leadership, advances have been made in the development of sound marketing practices for Pennsylvania crude products. Commendable work by the trained field staff, under his guidance, has resulted in stricter policing to prevent misuse of the association insignia guaranteeing the quality of 100 percent Pennsylvania oil.

## New Form for East Texas Gasoline Buyers

MEETING objections of refiners who are parties to pending court suits in which the oil code is attacked, the Petroleum Administrative Board has developed a new form of contract to be used by purchasers and sellers of gasoline in the East Texas area to cover gasoline supplied from current production, it was announced August 15 by Oil Administrator Harold L. Ickes.

Each form requires that the seller will not process hot crude oil or produce gasoline in excess of the gasoline allowable. A provision is included that "in the event vendor cannot obtain sufficient crude oil at the current market price from which to manufacture the gasoline herein sold, then in that event, vendor shall be obligated to furnish only that gasoline which he is able to produce from his supply of crude oil or that which is furnished him by the vendee or his agent at the current market price."

It was announced that the Department of Justice has ruled that the form of contract is not contrary to the anti-trust laws.

## Institute Invited to Los Angeles in 1935

A RESOLUTION to invite the officers, the board of directors and the members of the American Petroleum Institute to hold the 1935 annual meeting of the institute in Los Angeles has been adopted by the board of directors of the California Oil and Gas Association.

Acceptance of the invitation was urged on the grounds that an important segment of the official personnel and the membership lives in California, that eight years have passed since an annual institute meeting was held here, and that the California fields, which supply nearly 20 percent of the total crude production of the United States, are worthy the close study of the entire industry.

## Cooperative Work on Testing Emulsions

INTERNATIONAL cooperative work has been inaugurated recently on the testing of emulsions. This work grew out of the 1933 World Petroleum Congress held in England, in which a number of papers were given and discussed on methods of testing bituminous emulsions. Discussion by the various interested parties brought out the possibility and desirability of international work on standardizing test methods, and as the result of an invitation to cooperate in the work, the A.S.T.M. is taking an active part through its Committee D-4 on Road and Paving Materials, immediate jurisdiction of the work being in the province of Subcommittee B-17 on Emulsion Tests, headed by Mr. J. S. Miller, Jr., of the Barber Asphalt Company.

Col. E. G. Wace, chairman of the Roads Emulsion and Cold Bituminous Roads Association, Ltd., of England, has taken a leading part in the work. He recently forwarded a sample of a bituminous emulsion, with an outline of the tests to be used, to those participating.

The tests to be conducted upon the sample cover the following properties: water content; homogeneity or spraying quality; viscosity (Engler); lability in use; and resistance to low temperatures. The samples received here have been tested by two members of Subcommittee B-17 and it is expected that a compilation of the results obtained by the various cooperators will prove of considerable interest.

Tests included in specifications for emulsified asphalt being submitted to the society for approval as tentative, involve viscosity (Saybolt); residue by distillation; settlement (five days); demulsibility; sieve test (homogeneity or spraying quality); stone coating test; tests on residue by distillation: penetration at 25 C., solubility in $CS_2$, ash, ductility at 25 C., specific gravity at 25 C.


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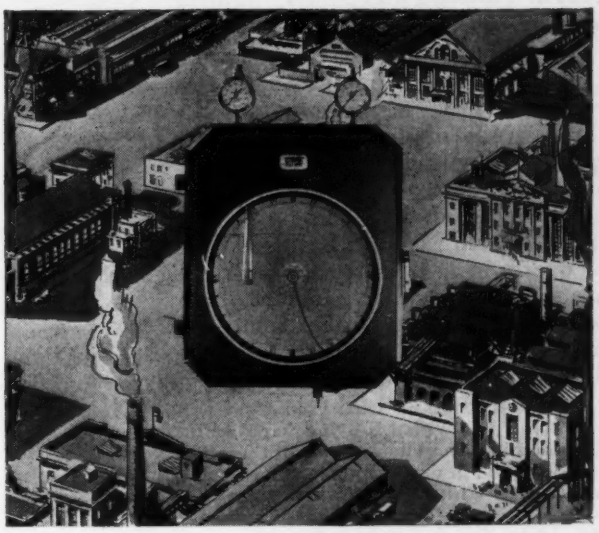

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In fuel to the extent of $324.00. In raw materials to the amount of $5,220.00. Through increased production to the extent of $6,384.00. Total, $11,928.00. The cost of the System was only about $4,000.00.

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## A.S.T.M. Annual Meeting; Detroit

THE A.S.T.M. annual meeting for 1935 will be held in Detroit during the last week in June; this decision having been reached by the executive committee at its last meeting. In addition, the Third Exhibit of Testing Apparatus and Related Equipment, will be held concurrently with the meeting. The first regional meeting ever held by the society was in Detroit, March, 1930, and in 1932 the Detroit District Committee headed by past- president, F. O. Clements, sponsored a successful local meeting and technical program.

The policy of the A.S.T.M. with respect to annual meetings is to hold meetings alternately in the East and in some Mid-West industrial center. Exhibits, sponsored by A.S.T.M., featuring testing instruments, machines and related equipment and research apparatus are held in conjunction with the annual meeting in the odd years. Successful exhibits, stressing apparatus developments in the fields of work in which the A.S.T.M. is active, were held in Chicago in 1931 and 1933.

## Pressure Vessel Code Available

THE Joint A.P.I.-A.S.M.E. (American Petroleum Institute-American Society of Mechanical Engineers) Code for the Design, Construction, Inspection, and Repair of Unfired Pressure Vessels for Petroleum Liquids and Gases is now available at the American Society of Mechanical Engineers, 29 West 39th Street, New York. The price is $1.00 per copy, with liberal discounts on quantity orders.

This code represents the culmination of several years of intensive work on the part of the Joint A.P.I.-A.S.M.E. Committee and of the groups that have cooperated with it, particularly the Subcommittee on Unfired Pressure Vessels of the Committee on Standardization of Refinery Equipment of the Division of Refining of the American Petroleum Institute.

This first edition of the code covers both welded and riveted vessels for operation at gage pressures above 15 pounds per square inch and at temperatures not over 1000°F. It is subdivided into four sections as follows: Section W—Fusion-welded Unfired Pressure Vessels; R—Riveted Unfired Pressure Vessels; I—Inspection, Repair, and Allowable Working Pressures for Unfired Pressure Vessels; S—Material Specifications.

The membership of the Joint Committee is: Walter Samans, Atlantic Refining Company, chairman; R. P. Anderson, secretary.

American Petroleum Institute: A. J. Ely, Standard Oil Development Company; K. V. King, Standard Oil Company, California (P. D. McElfish, alternate R. C. Powell, The Texas Company; Walter Samans, The Atlantic Refining Company; and T. D. Tifft, Sinclair Refining Company.

The American Society of Mechanical Engineers: R. E. Cecil, William B. Scaife & Sons Company; E. R. Fish, The Hartford Steam Boiler Inspection and Insurance Company; D. S. Jacobus, Babcock and Wilcox Company; T. McLean Jasper, A. O. Smith Corporation; and James Partington, American Locomotive Company.

## Moorhead Secretary Penn Grade Association

J. E. MOORHEAD, advertising manager for Rio Grande Oil Company (Sinclair Refining Company subsidiary), Los Angeles, has been elected by the board of directors of Pennsylvania Grade Crude Oil Association, to succeed T. O. Grisell as executive secretary of the association. He will assume his new duties October 1. Moorhead, until July 1, 1934, was publicity director for the association, resigning at that time to accept his present position with Rio Grande Oil Company.

## September Crude Oil Allowable

A DECREASE of 107,600 barrels daily in the national allowable production of crude oil was authorized today by Secretary of the Interior Harold L. Ickes, the Oil Administrator, in fixing the September total of 2,341,700 barrels daily, as compared with 2,449,300 barrels for August. The allowable becomes effective at 7 a.m. September 1.

This decrease resulted primarily from the normal seasonal decline in gasoline consumption as well as from an expected drop in gasoline demand due to the drought. An additional fact tending to lower the crude oil allowable in September was a necessity for reducing excessive gasoline inventories which are still considerably above a sound economic level.

Virtually all of the states shared in the decrease in the national crude oil allowable, no state receiving an increase. The three leading producing states, Texas, California, and Oklahoma, were reduced 32,900, 33,200, and 19,100 barrels from 1,001,300; 490,200, and 480,100, respectively. Other reductions were as follows: Arkansas, 1100 to 29,300; Illinois, 500 to 12,000; Indiana, 100 to 2200; Kansas, 10,500 to 120,700; Kentucky, 200 to 11,500; Michigan, 3900 to 29,300; New Mexico, 900 to 45,800; New York, 500 to 10,100; Ohio, 800 to 12,000; Pennsylvania, 2300 to 38,400; West Virginia, 500 to 11,100; and Wyoming, 1100 to 33,900. Allocations for Colorado, Louisiana, and Montana remained unchanged.

## troleum Division
## merican Chemical Society

AT the meeting of the Division of Petroleum Chemistry to be held in Cleveland, September 10 to 14, a number of interesting and valuable papers will be presented, and which will later be published in the society's journal, "Industrial and Engineering Chemistry." The following program of papers has been announced: The Isolation of a Nonanaphthaline from an Oklahoma Petroleum, by Joseph D. White and F. W. Rose, Jr.; The Critical Solution Temperatures of Some Hydrocarbons in Sulfur Dioxide, by R. T. Leslie; Knocking Characteristics of Hydrocarbons, by W. G. Lovell, J. M. Campbell and T. A. Boyd; Inhibitors in Cracked Gasoline, III, Storage Stability as Related to Induction Period and Critical Oxidation Potential, by C. G. Dryer, J. C. Morrell, Gustav Egloff and C. D. Lowry, Jr.; The Effect of Alkyldisulfides and Sulfur on the Color Stability of Gasolines, by Gustav Egloff, J. C. Morrell, W. L. Benedict and C. Wirth, III.; The Deterioration of Absorption Oils in Service, W. E. Kuhn and J. F. Collins, Jr.; A Method for Evaluating the Viscosity Temperature Characteristics of Oils, by W. B. McCluer and M. R. Fenske; The Molecular Weight of Cracked Distillates, by Ogden Fitzsimons and E. W. Thiele; The Specific Heat of Liquid Pure Hydrocarbons and Petroleum Fractions, by L. P. Gaucher; Efficient Small-Scale Fractionating Equipment, by C. O. Tongberg, D. Quiggle and M. R. Fenske, and An Apparatus and Conversion Chart for Routine High Vacuum Distillation of Oils, by K. M. Watson and Charles Wirth, III.

## Determining Helium In Natural Gas

AN improved apparatus and method for rapid and accurate determination of the helium content of natural gas and gaseous mixtures derived from that source have been developed in the Cryogenic Laboratory of the United States Bureau of Mines, Department of the Interior. A complete description of the apparatus and procedure for analysis has been made generally available to the public in a recently issued report of the bureau under authorship of C. C. Anderson, associate chemist, U. S. Bureau of Mines Cryogenic Laboratory, Amarillo, Texas.

These improvements are the result of technical investigations which are a part of the bureau's helium program, directed toward reducing costs of supplying all of this non-combustible gas used in the lighter-than-air-craft of the army and navy and toward assuring adequate supplies of this phenomenal war mineral.

The quantitative determination of helium in a gaseous mixture is accomplished by the removal of the other constituents from the sample, either by chemical or physical means, leaving only the helium which can be measured directly.

When cooled to liquid-air temperatures, activated coconut charcoal absorbs large volumes of the principal constituents of natural gas, such as nitrogen, methane, other gases of the hydrocarbon series, carbon dioxide, and oxygen, if the latter is present in the sample but does not absorb detectable quantities of helium, under the conditions prevailing in the analytical apparatus. The thousands of analyses made in the Cryogenic Laboratory indicate that in natural gases of this country hydrogen and neon are either absent or occur in such small percentages as to be absorbed readily by the quantity of charcoal usually employed.

Essentially, the apparatus consists of a condensation tube, two charcoal tubes, a Topler mercury pump, and a burette. All of these parts and the necessary mercury traps, stopcocks, and manometer are welded together, forming a vacuum-tight apparatus. Accessory equipment includes a spectroscope, spark coil, six-volt storage battery, a balance for weighing, a gasometer or equivalent for holding the sample before analysis, vacuum flasks for holding liquid air, and pumps or other

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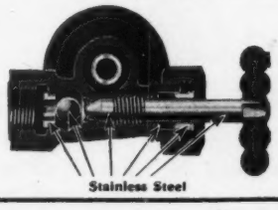

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devices for supplying compressed air and creating a vacuum to operate the Topler mercury pump.

For routine work in the Cryogenic laboratory, some modifications of the more elaborate apparatus have been made, and since there is always the possibility of finding other rare gases associated with the helium in the samples analyzed in connection with the helium-resources survey, a wave-length spectrometer is used in order that those gases may be identified.

After becoming familiar with the apparatus, an analyst can complete determinations in 15 to 30 minutes, depending upon the volume of helium to be pumped out of the apparatus, and with ordinary care results can be duplicated within close ranges. For samples having one percent or less of helium, check determinations should not vary more than 0.01 percent. Check results for samples having from one to two percent helium should be within $\pm$ 0.01 percent. Copies of I. C. 6796, An Apparatus and Method for the Determination of Helium in Natural Gas, may be obtained without charge upon request to the Information Division, U. S. Bureau of Mines, Washington, D. C.

## The Accounts of An Oil Company

**By H. G. Humphreys.** Cloth bound volume, 136 pages, price $2. Published by American Institute Publishing Company, Inc., 135 Cedar Street, New York, N. Y. For sale by Gulf Publishing Company, Houston, Texas.

This book was selected by the committee on publication of the American Institute of Accountants as one of its principal titles for this year. It was published through recognition that great changes have taken place in the methods of producing and refining oil and that there have been accompanying changes in the methods of accounting.

Some of the material was published during 1933 in *The Journal of Accountancy* in the form of series of articles on various phases of the petroleum industry. Those articles have been amplified and much new matter has been added.

The book is divided into eight chapters, taking up in sequence the following subjects: balance sheet and general accounts, departmental investment and income, forecast of income, oil and gas properties, natural gasoline department, refinery department, lubricating department, and marketing department.

## Transactions A.I.M.E. (1934)

**Petroleum Development and Technology.** Cloth bound volume, 465 pages, price $5. American Institute of Mining and Metallurgical Engineers, 29 West 39th Street, New York, N. Y.

This book, recently off the press, contains papers presented before the division at Los Angeles in September, 1933, at Dallas in October, 1933, and at New York in February, 1934.

The papers are divided into five chapters, covering production engineering and engineering research, petroleum economics, stabilization data, production statistics on domestic and foreign fields, and developments in refinery engineering during 1933.

The chapter containing statistical information on production is worthy of particular mention, as it presents the statistics in a new and more comprehensive form than previous publications of the organization.

Another new feature of the volume is a table showing the consumption of petroleum throughout the world in 1931, 1932, and 1933, included in the chapter on petroleum economics.

The volume represents a valuable and authoritative compilation of engineering and statistical data on petroleum, appealing to engineers, economists, geologists, executives, and manufacturers.

# ▼ PLANT ACTIVITIES ▼

**Dewaxing Plant:** Standard Oil Company of Louisiana, Baton Rouge, is completing erection of a Sharples Bari-Sol centrifugal dewaxing installation, Max B. Miller Co., Inc., licensor, and this installation using mixture of ethylene dichloride and carbon tetrachloride as solvent.

**Purchase:** Frank Weber and Al Clark, Farmington, New Mexico, have purchased a refinery at Aztec, New Mexico, to move equipment to Farmington, recondition and enlarge.

**Refinery:** The Mexican government is reported considering establishment of a $5,000,000 petroleum refinery at Mexico City. Indefinite maturity.

**Pipe Line:** Standard Oil Company of California to build 108 miles of 12-inch pipe line, including five new pump stations and reconditioning of two operating stations. Line to cost $4,000,000 and to extend from Rio Bravo pump station, Kern County, and join with present lines from Kettleman Hills to Estero Bay.

**Merge:** Bell Oil & Gas Company has merged with Altitude Petroleum Company, retaining the Bell name. Albert Finston is president, Jesse Finston, first vice president. I. A. Anson and M. L. Dreese of Altitude Petroleum Company, become vice presidents in charge of sales for the new company, which owns refineries in Oklahoma and Kansas.

**Operating:** Lomita Gasoline Company has started operating its new gasoline absorption plant in the Mountain View field, Kern County, California. Constructed by F. H. Garbutt and designed by J. A. Campbell.

**Sold Plant:** Gulf Production Company has sold its three-unit natural gasoline plant, Breckenridge, Texas, to Lone Star Gas Company. Purchaser has shut the plant down.

**Enlargement:** Mohawk Oil Company, Fruitvale, California, has enlarged its refinery through the addition of a new 2000-barrel distillation unit.


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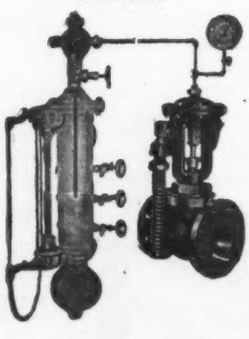

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**Purchase Unit:** McMurrey Refining Company, Arp, Texas, R. J. McMurrey, president, has purchased from Kettle Creek Refining Company, El Dorado, Arkansas, a cracking unit and equipment is being moved to install at the Arp refinery.

**Enlarge:** M. M. McCallem Refining Company, Huntington Beach, California, is adding a new unit to its road oil and asphalt plant. Estimated cost $10,000.

**Coking Process:** Lubrite Refining Corporation (Socony-Vacuum Corporation), St. Louis, Missouri, has awarded contract to H. A. Brassert & Company, Chicago, for a three-unit installation of the Knowles residuum treating process. The Knowles ovens with coke handling facilities, as well as pipe still and fractionating equipment are included. The latter will be built by J. P. Devine Corporation.

**Carbon Black Plant:** Ben Ashe and associates have been issued permit by the Oklahoma Corporation Commission to establish a carbon black plant three miles from Guymon in the Oklahoma Panhandle. Estimated cost $500,000. Gas from the undeveloped Texas County field.

**Purchase:** C. L. & W. Refining Company, Inc., Gladewater, Texas, has purchased Carnation Refineries, Inc., 1500 barrel skimming plant at Gladewater. Plant has been operating since February 1, 1933.

**Sale:** .Lawrence Gasoline Plant, Inc., Dallas, has sold its natural gasoline plant in the Kilgore section of the East Texas field to the Frontenac Pipe Line Company, a new subsidiary of McColl-Frontenac Oil Company, Ltd., owner of refineries at Montreal and Toronto, Canada. Frontenac Pipe Line Company also acquired the crude gathering system of the Mil-Hil Corporation, Dallas, that is connected to about 25 wells in the Kilgore area, and will mix the crude and natural gasoline in making deliveries via tank cars to the Canadian plants.

**Sale:** Tall Pines Natural Gasoline Corporation, Tyler, Texas, has sold its natural gasoline plant in the Kilgore section of the East Texas field to G. L. Culver, Gladewater operator, who also operates Linzie Refining Company and Culver Refining Company plants. Culver is reported to have paid $50,000 cash and assumed all outstanding obligations in acquiring the above gasoline plant.

**Solvent Plant:** Vacuum Oil Company is installing a small unit of the Chlorex solvent refining process at Olean, New York. The company is now operating its Duo Sol process at the Paulsboro, New Jersey, refinery.

**Purchase:** Phillips Petroleum Company has purchased from Bell Oil & Gas Company its well-pressure absorption type gasoline plant in western Wheeler County, Texas, on the A. R. Evans tract, NW Section 51, Block 24, H&GN Survey, and will enlarge capacity.

**Booster Plant:** Phillips Petroleum Company is planning a large gas booster plant in western Wheeler County, and has purchased a 40-acre site in NW SW Section 48, Block 24. The station will be hooked up to the present system of trunk lines transporting gas and natural gasoline to the company's plants in Gray County.

**New Equipment:** Freedom Oil Works Company, Freedom, Pennsylvania, is erecting a new 550 horsepower boiler and chain grate stoker. Boiler erected by Union Iron Works, Erie, Pennsylvania.

**Pipe Line:** Middlesex Pipe Line Company, an interest of Sun Oil Company, plans construction of a new welded-steel pipe line from the refinery of Sun Oil Company to bulk terminal on the Passaic River, Newark, New Jersey, at an estimated cost of $475,000. The line will include booster pumping stations and will run from Marcus Hook to Washington's Crossing, Pennsylvania, thence across the Delaware River, through New Jersey to the terminus, a distance of about 100 miles.

**Carbon Dioxide Plant:** Liquid Carbonic Corporation, 41 Mills Street, Toronto, Ontario, Canada, plans construction of a plant for the manufacture of carbon dioxide and carbonic ice. Estimated cost $50,000.

**Laboratory:** Gulf Refining Company, Gulf Building, Pittsburgh, plans erection of a group of three buildings to be used as a research laboratory at Harmarville, Pennsylvania. H. H. Henderson, Pittsburgh, chief engineer. Estimated cost $150,000.

# Don't Guess—Know!

## There Is Only One Way To Know For Sure
## Use Fisher Alignment Capacity Charts

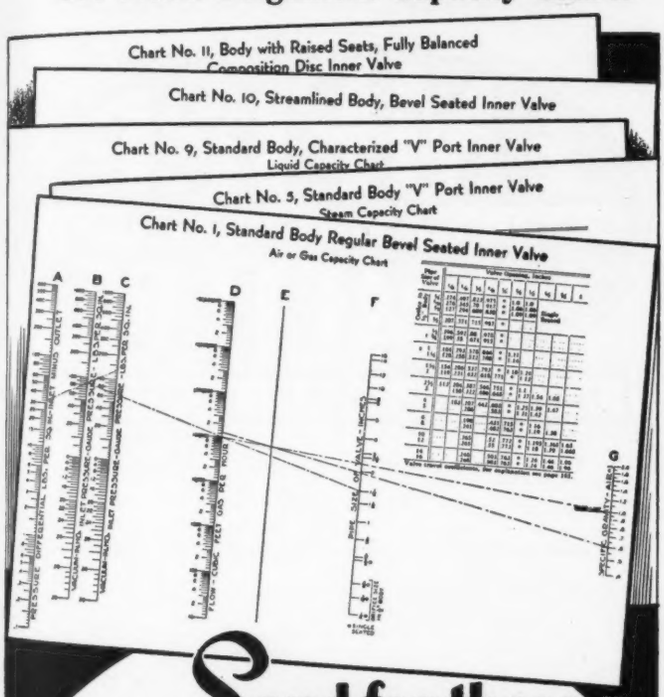
Chart No. 11, Body with Raised Seats, Fully Balanced
Composition Disc Inner Valve
Chart No. 10, Streamlined Body, Bevel Seated Inner Valve
Chart No. 9, Standard Body, Characterized "V" Port Inner Valve
Liquid Capacity Chart
Chart No. 5, Standard Body "V" Port Inner Valve
Steam Capacity Chart
Chart No. 1, Standard Body Regular Bevel Seated Inner Valve
Air or Gas Capacity Chart

## Send for these Capacity Charts

FISHER

Don't guess—Know! There is only one way to correctly determine the size of an automatic regulator for a given control job, or for determining the flow through a given size regulator—use the new FISHER Alignment Capacity Charts. These Charts cover various types of valve bodies and inner valve constructions. You can determine pipe size of regulator, pressure drop, pressure differential, flow, or gravity correction by using this most detailed and accurate up-to-date information ever published on this subject, now available after years of actual testing by FISHER engineers. You can save money and improve control by accurately sizing automatic regulators for your control problems.

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**have been used for steam boilers for 25 years and are now being used by leading oil companies for indicating oil levels in tanks, towers, stills, etc.**

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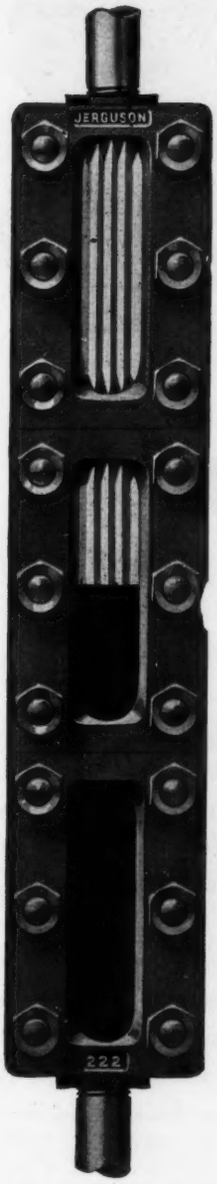


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**SOMERVILLE, MASS.**

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# LABOUR
## CHEMICAL VALVES

For more than four years LaBour Chemical Valves have been making good in many a process plant, including a considerable number of refineries. The basic design of these valves, tested by years of actual service, is retained in the new models now offered by LaBour, but the new ones include a number of improvements and refinements making them even more serviceable, and more convenient to install and use.

LaBour Chemical Valves of standard materials will handle most of the acids (outside the chlorine group) and practically all caustics. On special order valves can be supplied for almost any specified liquid.

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**Originators of the Self-Priming Centrifugal Pump**

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**Oil men everywhere like the Empire Meter. They like it because it is accurate, dependable, and economical. The Empire is the only meter of the oscillating piston type which is made in all sizes up to 6 inches. It is also the only BALANCED-PISTON meter.**

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# OIL METERS
NATIONAL METER COMPANY

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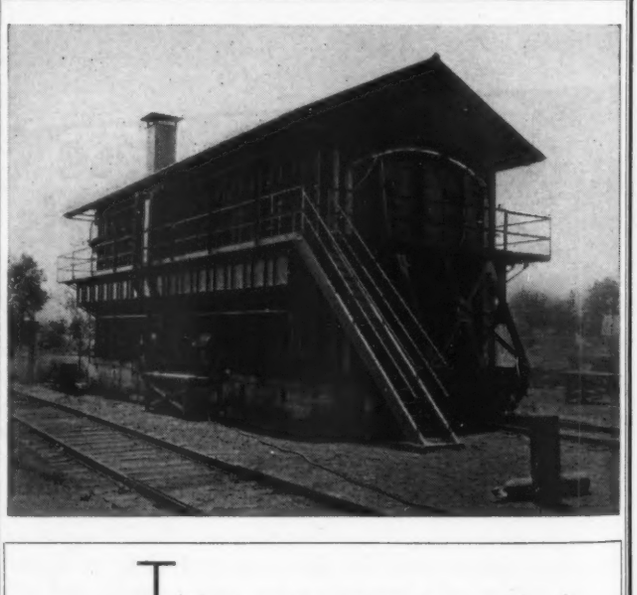

THIS Annealing Oven is capable of stress-relieving vessels up to 11′ diameter and 100′ in length, and with our X-ray Department places us in position to supply Class I and Class II Code Vessels to meet your requirements.

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## J. P. DEVINE MFG. CO., Inc.

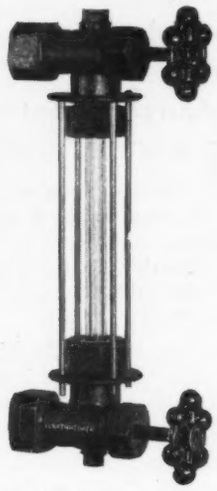
Subsidiary of Mt. Vernon Car Mfg. Co.
MOUNT VERNON, ILL.

# New Equipment for the Modern Plant

## Liquid Gauge

**THE LUNKENHEIMER COMPANY**

The Lunkenheimer Company, Cincinnati, Ohio, announces improvements and changes in design in its No. 1183 automatic liquid gauge. All internal working parts, stem, gland, retaining ring, stuffing box washers and ball checks are now made of stainless steel to provide maximum resistance to corrosion and to reduce sticking. The new pressure rating is 350 pounds gas-liquid pressure at 450°F.



*Lunkenheimer Gauge*

The new gauge has been tested and proved in actual field service. It is adapted for use on pressure tanks, gravity tanks and other reservoirs handling liquids which attack brass or bronze; where a non-corrodible, non-sticking gauge is essential. Sturdy construction is provided to assure dependability and safety; automatic closure of ball checks in both upper and lower valves in the event of glass breakage; non-corrodible and non-sticking working parts; easy and safe replacement of new glasses; cone shape packings to prevent leakage around glass, and minimize glass breakage hazard; standard pipe plug in lower valve body for drain connection and provided with a protective coating that retards rust and improves appearance.

## Voltage Booster

**WESTINGHOUSE ELECTRIC & MFGR. COMPANY**

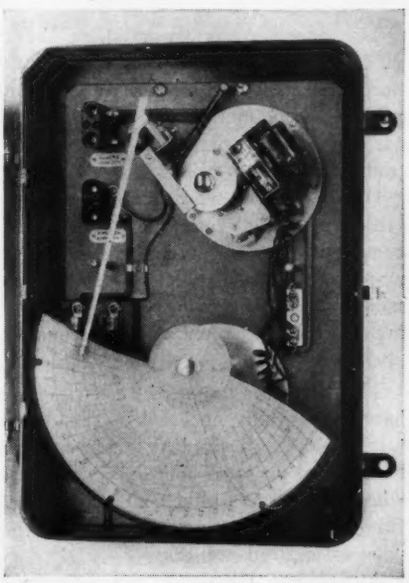
Westinghouse Electric & Manufacturing Company, East Pittsburgh, Pennsylvania, has announced a low priced, pole-mounted, booster which automatically changes a feeder's voltage either five percent or 10 percent in a single step. This new Type B Feeder Voltage Booster is particularly suitable for maintaining proper voltage for rural lines, isolated loads, or distribution circuits where the load changes once or twice daily from very light to practically full load. Single phase 60 cycle boosters range from 1½ kv-a. at 2400 volts up to 50 kv-a. at 6900 volts. Three phase three-wire circuits require two units connected in open delta; three phase four-wire circuits require three units in star. The booster is ordinarily controlled by line current, but can be operated from line voltage, if desired. Also, a special design is available for bucking instead of boosting voltage.

## Operation Recorder

**THE BRISTOL COMPANY**

The Bristol Company, Waterbury, Connecticut, is offering an electrical operation recorder, which provides a long record on a fast moving round chart. This instrument, instead of making one circular record, draws a spiral curve starting at the outer edge of the chart and gradually moving toward the center. This spiral record is accomplished by a telechrom cam which continuously resets the record making ment.

This instrument is meeting favor in time-study work, since it provides an open record with the easy filing and ready reference characteristics of the round chart. One form of the instrument is fitted with a chart driving clock, making one revolution per hour and with a separate telechrom motor driven cam making one revolution in eight hours. The resulting record is a spiral curve covering eight rotations of the chart and with approximately one-fourth inch spacing of the record line.



*Bristol Recorder*

The mechanism which records the occurence of an operation is a small electro-magnet which is energized when an external circuit is closed. The movement of this magnet actuates the pen arm and moves the inking pen approximately one-sixteenth of an inch. The movement is housed in a moisture-proof rectangular case.

## Integral-Furnace Boiler

**THE BABCOCK & WILCOX CO.**

The Babcock & Wilcox Company, 35 Liberty St., New York, announces a new boiler unit, designed to provide a completely coordinated unit comprising a two-drum boiler, a water-cooled furnace, burners for liquid, gaseous, or pulverized solid fuels, and, when needed, a superheater, economizer, and an air heater. This unit is applicable in industrial plants where operation at high nominal ratings with high final steam temperatures, and where high efficiency is desired. The arrangement of the boiler proper, with inclined tubes entering an upper and a lower drum, and with the furnace paralleling the drums and separated from the rows of tubes by a furnace wall, makes its installation advantage where headroom is limited.

The furnace is water-cooled to assure low maintenance costs and minimum outage for repairs and replacements. This feature is shown by the arrangement of the cooling-water tubes, the simplicity of the supporting structure, the use of but two headers, and the necessity for but one small section of steel casing. The two side walls are of Bailey stud-tube construction employing tubes, suitably spaced, with the spaces between the tubes filled with plastic refractory material secured in place by studs welded to the tubes. The floor is water cooled. The cooling tubes are straight and are covered with standard Bailey bare-metal blocks. The tubes of the floor and outer sidewall connect to a common header in such a manner that positive circulation is assured through the floor and the wall. The rear wall, or refractory construction, is protected by a row of bare water-cooled tubes. Pro-

tection of the boiler tubes and superheater is obtained by a slag screen, formed by the first two rows of boiler tubes. These tubes are offset or staggered, and are spaced on wider centers than those directly behind them to cool the molten sticky slag, without, however permitting the slag to bridge across the tubes and thereby restrict the flow of gases. The unit is suitable for either wet or dry ash removal.

The boiler tubes are arranged in three passes, and are so spaced as to secure maximum heat transfer consistent with a draft loss that is sufficiently low to permit operation with natural draft up to fairly high ratings. The tubes in the first pass are 3¼ inches in diameter, and the diameter of those in the succeeding passes is two inches.The tubes are on alternate wide and narrow centers to facilitate their renewal. All tubes can be cleaned with standard turbines, and the outside of the tubes can be cleared of all ash deposits with a minimum number of soot blowers.

The drums are of fusion-welded or, if desired, riveted construction. The lower drum rests upon cast-iron saddles, which, in turn, are suported by footings. It is anchored by the saddle near one end, but is free to move at the other end. The upper drum is supported by beams carried on corner columns, and, in the larger sizes, by auxiliary guides and supports secured to the buckstays and rear corner angles. It is equipped with the necessary steam-and-water separator to assure the delivery of dry steam. Steam enters the dry pan at the front of the boiler where the water is comparatively quiet, passes over the length of the dry pan, and is taken away from the rear of the drum to the superheater.

## Beryllium Copper Tools

**STANLEY TOOLS**

Stanley Tools, New Britain, Connecticut, has placed on the market a new line of non-sparking tools made from Anaconda beryllium copper. This alloy, which contains over 97 percent copper, can be hardened, toughened and strengthened by combined working and heat treating until its physical qualities exceed those of any other non-ferrous metal. Beryllium copper is non-magnetic and will not give off sparks when struck against other metals or materials. It is immune to rust and highly resistant to a wide variety of corrosive agents.

Stanley non-sparking beryllium copper tools are made from wrought and heat-treated metal which provides them with strength and toughness. The line includes hammers, chisels, scrapers, sledges, pinch bars, screw drivers, wedges, fork tips and other items.

## Putty

**B. F. GOODRICH RUBBER COMPANY**

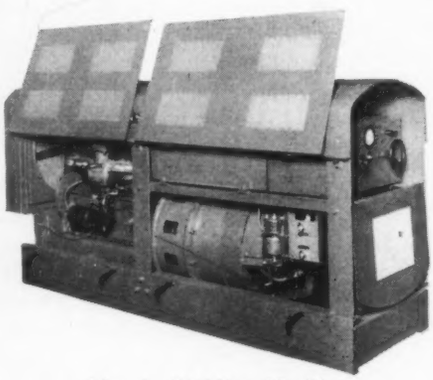
B. F. Goodrich Rubber Company, Akron, Ohio, has announced a new type putty which contains rubber. It is similar in appearance and consistency to painter's putty, and the manufacturer claims several advantages for it.

The putty requires no mixing, since it contains little oil. It is resistant to corrosive chemicals and fumes, and because of its rubber content offers a high resistance to moisture. It also adheres well to steel surfaces. The putty is called "Plastikon" and is marketed in 15-pound, 75-pound and 225-pound cans for commercial users, and in pint cans for small jobs.

## Welding Machine

**LINCOLN ELECTRIC COMPANY**

Lincoln Electric Company, Cleveland, Ohio, has announced a new close coupled gas engine driven "Shield-Arc" welder of 300-ampere, 40-volt capacity. Due to close coupling features, the new unit is much more compact and lighter in weight than previous models.



*Lincoln Welding Machine*

The generator is mounted on the engine housing with the drive lined up on hardened steel and driven on cushioned rubber. This method of coupling takes care of misalignment and reduces vibration.

Due to reduced vibration, the engine operates at a high efficiency and gasoline consumption is lowered, according to the manufacturer. A special idling device which cuts the engine speed when the arc is broken further assures economical operation of the six-cylinder engine.

The welder is mounted on a rigid steel base with totally enclosed steel side curtains. Where desired the complete unit is mounted on small factory portable steel wheels, or large six-inch face steel wheels for outside use, or on two or four wheel pneumatic tired trailers.

A uniform generator current permits use of a higher average current with greatly increased welding speed. The capacity of the new welding machine is 60 percent higher than its predecessor and it is rated at 40 volts. According to the manufacturer, the "Shield Arc" welder has 10 percent higher efficiency at full loads and still more on overloads, even though it is of much greater capacity.

Other features of the "Shield Arc" welder include dual control of welding heat, sparkless commutation, handy-height controls, and arc welded steel construction.

"Lincontrol," a new remote current control device which enables the operator to adjust the current by tapping the electrode on the work, is supplied as optional equipment. No additional cables or rheostat are carried by the operator.

## Direct-Current Motors

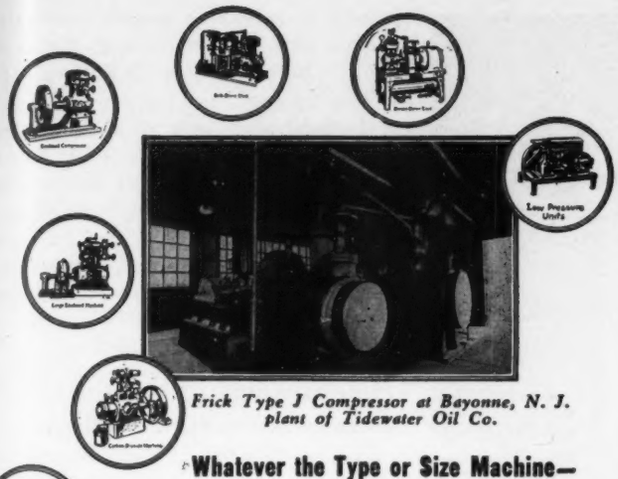
**GENERAL ELECTRIC COMPANY**

General Electric Company, Schenectady, N. Y., announces a new line of direct-current motors, designed for those applications where dust, dirt, moisture, or other foreign matter is present in large quantities. The new motors are totally enclosed and fan-cooled, and are available in a wide variety of electrical and mechanical modifications—in sizes from ½ to 200 horsepower.

A system of dual ventilation is utilized. This together with water-tight conduit boxes, and labyrinth seals at the cartridge-type bearing housings effectively protects the working parts of the motor. An internal fan draws the warm air from the windings and core and circulates it around the totally enclosed interior of the motor so that it gives up its heat to the frame and end shields. An external fan draws cool air in through mesh openings in an outer end shield and directs it over the surface of the magnet frame and inner end shields. Close clearances between the end shields and the bearing housings, and grease seals along the shaft, prevent foreign particles from entering into the motor.

These new direct-current motors are especially suited for application in locations where obnoxious process-dusts prevail, and to such apparatus as stokers, conveyors, pumps, machine tools, sand screens and shakers. They are suitable, too, for outdoor use in mild climates.

In addition to the advantages of total enclosure, these fan-cooled motors offer, as compared with totally enclosed motors, a saving in space, greater output per pound of material, economy of operation, and interchangeability with most open motors of corresponding ratings.



*Frick Type J Compressor at Bayonne, N. J. plant of Tidewater Oil Co.*

**Whatever the Type or Size Machine—**

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**Refrigeration**

**Provides refineries with the dependable, well engineered cooling service that can be offered by a firm with half a century of refrigerating experience. Get literature and recommendations now: write**

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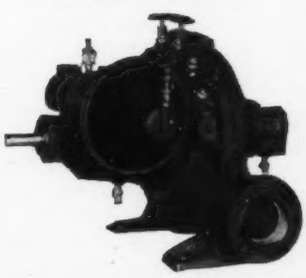
**STEAM TURBINES**

***For Longer Economical Trouble-Free SERVICE***



**Horizontal and Vertical Types**

**COPPUS ENGINEERING CORP.**

360 PARK AVE.
WORCESTER, MASS.

*Write for*
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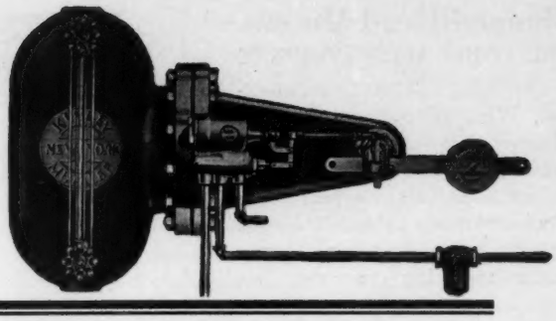

# New Features in Remote Control of Liquid Levels

Among the special features of this K & M Remote Control Liquid Level Controller is the precision pilot valve. This sliding type valve makes possible an ideal balanced pressure condition and assures sensitive control on high pressure or low.

A compensating device which prevents the main valve from making a complete movement from full open to full close, provides a fine throttling movement so that flow is in exact balance with requirements.

Refineries are daily demonstrating the superiority of this K & M Type Remote Controller and are materially reducing costs by its use.

Bulletin C-5 describes this and other types of K & M Liquid Level Controllers in detail.

**Send for this free "text book"**

**A 24 page 8½" x 11" bulletin on Liquid Level Control. Fully describing with applications, K & M Controllers of the internal or external float type, direct or remote controlled and illustrating special new features of this old established line of level control apparatus. Write for your free copy today.**

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*Main Office and Warehouse:*
**34 WEST 13TH ST., NEW YORK, N. Y.**
*Factory:* **Newark, N. J.**

**K and M**

**PRESSURE AND LEVEL CONTROL APPARATUS**

**K & M Products—VALVES: pressure regulating, air pilot operated, balanced, back pressure; LIQUID LEVEL CONTROLLERS; PUMP GOVERNORS; STEAM TRAPS**

## Flanged Head Motors

**THE LOUIS ALLIS COMPANY**

The Louis Allis Company, Milwaukee, Wis., announces the development of several types of flange head mountings for its line of electric motors. The flange is an integral part of the motor head and may be finished on either or both sides. Limitations in space for motor mounting on many applications such as machine tools, including lathes, drills, grinders, polishers, etc., make it desirable to eliminate motor feet and provide finished flange on motor head for end mounting.

This new type of motor is also used widely on close-coupled pump mountings.

## Safety Switches

**CUTLER-HAMMER, INC.**

Cutler-Hammer, Inc., Milwaukee, Wisconsin, announces a new line of explosion-proof safety switches for use in Class 1, Group D, hazardous locations. The line includes both single- and double-throw types in standard sizes up to 200 amperes capacity. The switch is a heavy, industrial duty Type A construction, with outside operating handle, mounted in a heavy weatherproof, semi-steel cast enclosure, complying with requirements of the national electrical code. A precision machined flange of required width, between the case and cover, assures proper cooling of any flame which might occur from an explosion within the case, so that outside gases will not ignite. Corrosion resisting bolts hold the cover firmly in place.

*Cuttler-Hammer Safety-Switch*

Two pipe threaded conduit holes are provided in the bottom of the case and pads at the side and top of the case allow for drilling other holes if needed. Provisions are made for padlocking the handle in either position—accommodates three padlocks in "off" and three padlocks in "on" positions to meet specific industry requirements. Finish is black Japan.

Specific applications for these explosion proof switches include such locations as distilleries, dry cleaning plants, petroleum refineries, bulk oil stations, filling stations, gas plants, spray painting plants, chemical works, paint, lacquer and varnish works, etc.

## Proportioning Unit

**D. W. HAERING & COMPANY**

D. W. Haering & Company, 3408 Monroe Street, Chicago, announces the development and introduction of the H-O-H Proportioning Unit, an automatic proportioning machine which makes the accuracy of the pitot tube principle practical.

The machine will feed liquid chemicals into liquid lines under varying conditions of pressure and in any amounts desired. The machine is entirely automatic. It starts, stops and feeds in proportion to flow. The only manual attention required is the recharging of the unit which may be accomplished in less than five minutes and the reagent tanks are made of sufficient size to require this operation only at long intervals.

The machine is finished in baked enamel and sells at a very low price setting new records of economy in the proportioning equipment field.

The unit is also easy to install as it is only necessary to drill and tap three-eights of an inch pipe line in order to install the pitot tubes. Once the tubes are installed it is possible to disconnect the machine and make all piping arangements or changes from time to time without interfering with the main flow of liquid.

## Disconnecting Switches

**GENERAL ELECTRIC COMPANY**

General Electric Company, Switchgear Sales Division, Philadelphia, announces a new line of indoor disconnecting switches, with ratings as high as 1200 amperes at 5000 volts, featuring silver-to-silver line-pressure contacts. The silver, applied to the contacts by a special process, produces a surface said to be of unusual wearing qualities, to eliminate troublesome oxidation and its inherent disadvantages.

The complete line includes hook-operated, single- and double-throw, double-throw double-blade transfer switches with half- or full-capacity blades, single-throw tandem transfer switches with full-capacity blades, and group-operated switches controlled manually or electrically. The single- or multi-pole switches are mounted on compound insulators and metal bases. The insulators are made of non-inflammable, arc-resisting Cetec cold-molded material and have a dry flashover of 25 KV.

## Diesel Engine

**THE GUIBERSON CORPORATION**

The Guiberson Corporation, Dallas, Texas, has introduced a line of Diesel engines for trucks and buses, oil well pumping and other industrial power requirements. The new line of industrial engines was the outgrowth of the company's success with its Diesel aircraft engine.

The special patented feature of the Guiberson engine is the synchronization of fuel supply, timing, and air supply so that all are controlled by one throttle, and proper proportions and relations of each automatically assured at all speeds, the manufacturer claims.

The engine has been perfected during several years of experimental work. A 7½-ton truck powered with this Diesel engine was driven all over the United States. A loaded truck of this size was driven recently from Dallas to Tulsa, a distance of 300 miles, with a full operating cost of $1.75, the manufacturer has announced.

A.S.T.M.-C.F.R. OCTANE RATINGS
Natural Gasoline and Gas Analysis
Using Podbielniak apparatus.
All other tests on Petroleum Products.
**Phoenix Chemical Laboratory**
"an exclusive petroleum testing laboratory"
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Electrically Driven Centrifuges
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PULMOSAN RESPIRATORS
Protection against DUST SPRAY MISTS SANDBLASTING
for EVERY NEED
WRITE for DETAILS
PULMOSAN SAFETY EQUIPMENT CORP.
176 Johnson St. Brooklyn, N. Y.

# Items About Manufacturers

**Wood-Moore Corporation,** Tulsa, announces opening of a branch at Corpus Christi, Texas, with H. G. Schiveley in charge. Arrangements have been made with General American Tank Storage and Terminal for terminal facilities through that company's new terminal recently purchased from the Corpus Christi Terminal Company. The company is starting immediately to enlarge its newly acquired terminal to take care of Wood-Moore Corporation's export and coastwise business. Wood-Moore Corporation own a refinery and natural gasoline plant recently completed at Banquette, Texas.

**Koalt Engineering Company** of Tulsa announce the opening of an office in Dallas, Texas, with Neff S. Cass in charge. The company is distributor in the Southwest for Key Boiler Equipment Company and Timken Steel and Tube Company products.

**Southwestern Engineering Corporation,** Los Angeles, is out of the hands of a receiver and is being operated by a new and strengthened organization under the name Southwestern Engineering Company under a reorganization plan perfected by stockholders. The company has been a prominent manufacturer of petroleum refining and natural gasoline equipment for many years. Officers of the new concern are: H. G. Miller, president and general manager; Geo. W. Clemson, vice president; Janet Rattray, secretary-treasurer. Jack Coulter has joined the company as its chief petroleum engineer in charge of sales of refining and natural gasoline equipment.

**The Borden Company,** Warren, Ohio, announces the appointment of Rodney E. Mathews as district manager of its southern territory. For some years Mathews has been connected with a large supply house in New Orleans, and is widely acquainted with the trade in the south. The Borden Company manufactures the Beaver line of pipe cutting and threading tools.

**The Meriam Company,** 1955 West 112th Street, Cleveland, Ohio, is enlarging its plant space and capacity, the new building to be used for oil and gas engine rebuilding, permitting the present buildings to be used for the welding division and for the manufacture of flow and pressure instruments.

**Republic Steel Corporation,** Youngstown, Ohio, announce appointment of Buhl Sons Company, Detroit, as a recent addition to the country-wide organization of Toncan Iron distributors. Complete warehouse stocks will be maintained at the Detroit distribution point.

**Maintenance Engineering Corporation,** Houston, has appointed B. I. Thorngren as sales engineer to be directly engaged in water conditioning work. Thorngren was formerly with Magnolia Petroleum Company in the technical service division.

**Mine Safety Appliance Company,** Pittsburgh, has established a warehouse branch at Houston, under the direction of Geo. J. Gruber, original representative in the Louisiana-Texas territory, and John B. Baird. The new branch office is located at 2018 Capitol Avenue.

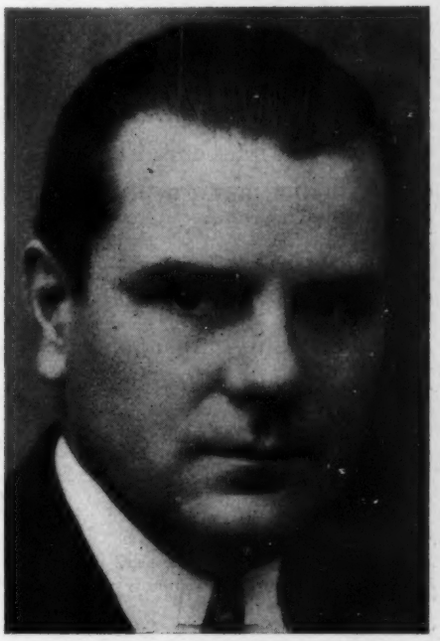
**Allegheny Steel Company,** Brackenridge, Pennsylvania, announces that Russell M. Allen, formerly Chicago district manager, has been promoted to the position of assistant general manager for the company.

**Pittsburgh Plate Glass Company,** Pittsburgh, has increased its distributing warehouse system to 71 units through acquisition of Montana Glass & Paint Company, Butte, Montana. The company recently started operating its window glass factory at Mt. Vernon, Ohio, giving employment to 400 men.

**John M. McDonald,** president of A. Y. McDonald Manufacturing Company, Dubuque, Iowa, died August 4. He was born in Dubuque, July 30, 1871. Following his education in the Dubuque public schools, he entered the business which his father had established in 1854. He became president of the company on the death of his brother, A. Y. McDonald, Jr., in 1923. Surviving him are his widow, Mrs. Mary A. McDonald, and two daughters, Mrs. Mary McDonald Pnapp and Mrs. Cornelia McDonald Higley; and two sons, both actively associated with the McDonald company—John M. McDonald, Jr., and William McDonald.

**Timken Steel & Tube Company,** Akron, Ohio, announces appointment of Koalt Engineering Company, 533 Mayo Building, Tulsa, as exclusive representative in Kansas, Oklahoma, Arkansas, Louisiana and Texas for the sale of steel and tubular products.

**Ralph M. Parsons Company** has been organized to carry on the consulting and contracting business which Parsons personally has been conducting for several years. Headquarters of the company is at Mt. Vernon, Ohio. While the organization will design and build all kinds of refining equipment, it will specialize in the design and construction of cracking units for small refineries to operate the Dubbs process under Universal Oil Products Company license and the rebuilding of existing units to operate under license contract and to insure highest efficiency.



**RALPH M. PARSONS**

Parsons has been engaged in commercial development work for many years. During the war he was active in aircraft engine designing, construction and operation. After he was released from active service in the navy as a reserve officer he was employed as aeronautical engineer to complete the design and construction of the Navy Department aeronautical engine laboratory at the Philadelphia Navy Yard. Since 1925 he has been engaged in design, construction and operation of refineries and refinery equipment. During this time a major portion of the work was in connection with the design and construction of cracking processes. He early recognized the

need for a small cracking unit, fully protected by legitimate license and specially designed to meet the requirements of those refiners having a limited supply of charging stock. Accordingly, three years ago he undertook to adapt and install a special cracking unit which would incorporate all of the performance characteristics of a larger unit with cost proportioned to the reduced capacity. Designing engineers, construction and operating men of Ralph M. Parsons Company all have had long practical experience in the refining industry and have all been long associated.

**Southern Alkali Corporation** this month started operation of its entirely new $6,000,000 plant at Corpus Christi, Texas, producing basic alkalis. As the first of a growing immigration of plants to serve industrial consumers of these products in the territory west of the line through Minnesota to Florida, the new plant offers quicker deliveries and more advantageous freight rates to 12 states east of the Rockies. In addition to rail facilities, the plant is located at tidewater with facilities for loading at its own docks for water shipment, both deep sea and through the recently opened intercoastal canal. Decentralizing of the basic alkali industry initiated by this producer not only means a new service deal for existing consuming industries affected, but undoubtedly will influence the development of many new consuming industries which can capitalize upon the advantages gained from a Southwestern source of supply. Initial production of soda ash will be approximately 500 tons per day.

**C. W. Stratford,** for the past two and a half years president and general manager of Alco Products International, 1 Rue Paul Cozanne, Paris, has resigned from the services of that company. C. H. Edmonds has been appointed to fill the vacancy caused by the resignation of Mr. Stratford.

**Jeffrey Manufacturing Company,** Columbus, Ohio, announces appointment of A. F. Brosky as special engineer attached to the general offices at Columbus.

**E. I. du Pont De Nemours & Company,** Wilmington, Delaware, through receivers sales, has purchased Mountain Varnish & Colors Works, Inc., Toledo, Ohio.

**C. W. Stratford** has re-established the Stratford Engineering Corporation, a company previously engaged in European and domestic oil refining equipment business, with offices in Kansas City, Missouri. He has recently resigned his position as president and general manager of Alco Products International of Paris, in which capacity he has served for the past three years.

J. A. Altshuler has resigned his position as technical director of Alco Products International of Paris as of September 1, 1934, to assume the position of an officer of Stratford Engineering Corporation.

**Foster Engineering Company,** 109 Monroe Street, Newark, New Jersey, has appointed Crawford Engineering Company, 295 Madison Avenue, New York, as its representative in the New York district. H. J. Fortier, formerly in the New York territory, is representing the company in the North New Jersey territory.

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LE HAVRE, France, received August 16 the first shipload of oil from the Iraq oil fields. The oil was routed through the 531-mile northern branch of the pipe line from Kirkuk, Iraq, to Tripoli, in French Syria. There a tanker picked up 14,586 tons and brought it to Le Havre, where it will be stored in tanks for use primarily by the navy.


# Phone 2-4432 Tulsa, Okla.

Is the telephone number of the Natural Gasoline Supply Men's Association.

If you desire information concerning equipment—Who makes it—Where it can be secured locally—call or write the Association, and the names of the manufacturers and their local distributors will be given to you.

This Association is organized to promote high ethical standards of business and to render a more efficient supply service and to promote a closer relationship among the supply men and the members of the Natural Gasoline Association of America.

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